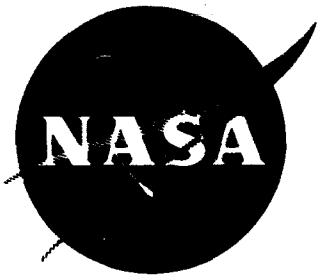

NASA CR-54771
MRB5010Q4

Quarterly Report No. 4

DEVELOPMENT OF THE DRY TAPE BATTERY CONCEPT

9 March 1966 to 9 June 1966

By

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Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS3-7624

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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SUMMARY

During this report period, we changed our aqueous static test method from one of current to one of voltage regulation. The new method more closely approximates the conditions present in dynamic tests.

Because of reduced gassing, an expanded magnesium anode is not a necessity when aqueous $MgBr_2$ electrolyte is used.

Electrolyte studies indicate that it is the amount of water, not acid, that limits the cathode reaction. Electrolyte added through the anode was utilized more efficiently than that added through the cathode mix.

The cathode mix compositon was altered in a number of ways in an attempt to reduce the amount of electrolyte necessary for cell operation. Neither a decrease in the percent Shawinigan acetylene black (SAB) used,nor parital replacement of SAB with a more dense graphite material appreciably changed cell electrolyte requirements. The current minimum is ca. 3 grams of electrolyte per gram of cathode mix. The incorporation of electrolyte salts ($AlCl_3$, $MgBr_2$) into a cathode mix increased wetting properties, but did not reduce the volume of water necessary to activate the cell. In some addition, active chlorine losses increased. Increased cell performance was observed when carbon fibers were replaced by paper pulp in the cathode mix.

Energy densities up to 88 watt-hr/lb have been obtained in static tests with $Mg/MgBr_2$/ACL-85® cells run at 2.0 volts and 0.11 amp/in^2.

Our initial liquid cathode experiment utilized a $Mg/MgBr_2/Br_2$ tape cell. An energy density of 39 watt-hr/lb was obtained at a very low coulombic efficiency (18%). Optimization of this system should result in the production of high energy densities.

Non-aqueous experiments in $LiClO_4$-methyl formate electrolyte have shown ACL-70® to be more useful as a depolarizer than ACL-85 or CuF_2. Energy densities in the $Li/LiClO_4$(MF)/ACL-70 system were improved by the reduction of the amount of carbon black in the cathode mix to 12%. Reproducibility problems appear to be the result of poor cell component contacts. An energy density of 144 watt-hr/lb has been obtained from the Li/ACL-70 system. Minimum electrolyte static cell holders were designed and tested for both aqueous and non-aqueous work. Data quality and reproducibility increased considerably.

A rolling current collector has been constructed for the dynamic test apparatus. Friction problems should be minimized with this system.

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I. INTRODUCTION

A. BACKGROUND

This work is a continuation of research performed under Contracts NAS3-2777 and NAS3-4168. During the initial program (NAS3-2777) the feasibility of the Dry Tape Concept was demonstrated using a divalent silver oxide-coated, porous polypropylene tape that was drawn between two current collectors. One current collector was a zinc block that also served as the anode. The cathode current collector was a thin silver plate. Electrolyte was supplied by a second tape prewetted with electrolyte and stored separately ("dual tape system"). The system was activated by bringing the two tapes together just prior to their entry into the current collector zone. The tapes were driven by a spring-wound motor, contained in a separate housing. Within this housing was an output metering and load section. Four such drive housings, together with 20 tape decks, were supplied to NASA for demonstration. In continuous operation, silver peroxide utilizations of over 90% were obtained at current densities of 1 amp/in.^{2}.

In the follow-on program (NAS3-4168), the "dual tape system" using the established battery couple, AgO/KOH/Zn, was replaced by a single tape configuration using a thin foil magnesium anode and cathode coatings containing nonconducting, higher energy organic (dinitrobenzene, picric acid, trichlorotriazinetrione) and inorganic (potassium meta-periodate) depolarizers. Efficient, high drain rate discharge of these cathode materials was achieved through use of the "thin plate tape electrode" configuration. The single tape configuration was optimized for the system, Mg/2M$AlCl_3$;0.5M HCl/KIO_4. Up to 80% KIO_4 utilization was achieved with a cell voltage of 2.2 volts at a current density of 0.5 amp/in.^{2} while in a moving configuration. In addition, to tape electrode configuration development, methods of electrolyte incapsulation and tape activation were devised. Also, techniques for supplying multiple cell voltage, parasitic drive, and continuous coated tape manufacture were developed. Early in this present contract, a new aqueous tape couple, based on magnesium and trichlorotriazinetrione (ACL-85®) in acidic electrolyte, was developed. Static cells operated routinely at 2.4 volts and 0.5amp/in^2. Cathode efficiencies were high (70-80%) but eratic. Energy densities of 300 watt-hr/lb of tape system (excluding electrolyte) were obtained. The variations in cathode efficiencies were traced to the use of an acidic electrolyte, a necessity for high current densities. At low pH, chlorine evolution occurred reducing the available active chlorine content. A change to a different acidic electrolyte helped, but did not completely resolve the problem. It was determined that cathode decomposition could be avoided if neutral electrolytes were used. In addition, anode gassing was reduced. Lower current densities, however, were necessary with the neutral system because of concentration

polarization effects. Sixty to seventy per cent cathode efficiencies were obtained at 2.0 volts and 0.05 amp/in^2.

As a result of preliminary anode, cathode and electrolyte studies, our non-aqueous research has emphasized the Li/$LiClO_4$-methylformate/ACL tape cells. These cells run at 3.2 volts and 0.05 amp/in^2. The major development problems associated with this system involve the difficulty in utilizing more than one active chlorine from the ACL molecule, and the low boiling point (32°C) of the electrolyte solvent.

B. FOURTH QUARTER OBJECTIVES

1. TASK I. High Energy Electrode Research

a. Cathode Research

(1) Optimize and standardize the ACL-85 cathode for neutral aqueous electrolyte systems.

(2) Test LiOCl as an oxidizing agent in both aqueous and nonqueous systems.

(3) Begin liquid cathode studies.

(4) Optimize the ACL-70 nonaqueous cathode.

b. Anode Research

(1) Investigate Be as an anode material.

(2) Investigate a 4:6 Li-Mg alloy.

2. TASK II. Tape Cell Evaluation

a. Electrolyte Studies

(1) Get quantitative electrolyte requirement data for both aqueous and nonaqueous systems.

(2) Study the incorporation of electrolyte salts into the cathode mix.

(3) Test aqueous acidic fluoride electrolytes.

b. Tape Cell Preparation

(1) Prepare uniform machine made tapes.

(2) Optimize the preparation of tapes for static tests.

c. Dynamic Testing

(1) Conduct dynamic electrolyte requirement evaluations

3. Supporting Research

a. Test Equipment Design and Improvement

(1) Redesign the dynamic apparatus cathode current collector.

(2) Redesign static test cells for minimum electrolyte studies.

(3) Design a pressurized cell for methyl formate electrolyte cells.

These objectives were attained, or significant progress was made toward all objectives except those concerned with anode research and dynamic testing.

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II. TASK I - HIGH ENERGY COUPLE RESEARCH

A. AQUEOUS ELECTROLYTE STUDIES

1. Anode Development

In the $Mg/MgBr_2(H_2O)/ACL-85$ cell, which was emphasized this quarter, anode gassing is less of a problem than it is with $AlCl_3$ electrolyte. Hence, a porous anode is not crucial. However, expanded magnesium (Exmet) anodes were found to be as good as either solid or punched magnesium anodes, (Table 1) and electrolyte addition was easiest through the expanded material. The use of Exmet Mg was continued this quarter.

TABLE 1

EFFECT OF TYPE OF Mg ON CELL DISCHARGE

Separator Wet Before Assembly - 2.5 ml
$MgBr_2$ Electrolyte - 0.1 amp/in^2 Discharge

Cell No.	Mg	Cathode Coulombic Efficiency %	Theoretical Capacity (amp-min)
84585-1	Exmet	51	29.7
-6	Punched	50	30.1
-8	Punched	43	32.6
-7	Solid	38	29.8

The coulombic efficiency in the tables in this report can be used as a figure of merit. The coulombic efficiencies are given relative to the amount of ACL-85 incorporated in the tape. Processing losses are not included. This causes the results to appear lower than in previous reports. This procedure is used, however, because of tape variation with time, especially with cathode mixes containing electrolyte salts.

2. Cathode Development - Constant Current Test Results With ACL-85 Cathodes

a. Introduction

During this quarter, we changed our static testing method from one employing constant current to one using constant

voltage. This was done to more nearly approximate dynamic tests.s While the results from both testing methods contribute to optimization of the cathode, the data are separated in this report to avoid confusion.

b. Tape Preparation

Tapes for static tests are prepared by mixing the dry components, adding tricloroethylene, and pressing the slurry on the separator. This general method was adopted because of its reproducibility, and because of its possible extension to continuous tapes for dynamic evaluation. The effects of the mixing variables, pressure and blotting variables in described in Section III (A) and in the table of data in the Appendix.

Preparation techniques were evolved during this quarter, starting with hand-cast tapes, which were air-dried, with and without pressure between polyethylene or blotting paper. After the discharge of ten batches of these tapes, the method was changed to accommodate a die and Carver press, and the method was standardized at 110 psi with blotting paper. This method gave tapes of uniform thickness with smooth surfaces. The pressure applied to the test cell during discharge was set with spring clamps and standardized at 4 psi. The mixing of the dry powders was accomplished by Waring Blendor for one minute, or for large batches, by a Patterson-Kelley twin shell blender with intensifier bar for 30 minutes.

c. Electrolyte Studies

(1) Position of Addition

It is possible to add electrolyte to the cell in two ways: through the expanded magnesium, or directly to the cathode. It is also possible, at least in the static test, to add electrolyte directly to the separator before assembling the cell. The results of these tests are shown in Table 2.

The results show that addition of electrolyte through the cathode requires excess electrolyte. Addition through the expanded Mg was as effective as addition to the separator before assembly. Electrolyte addition through the anode was adopted because it was reproducible, convenient, and applicable to dynamic tests without separating anode and cathode tapes.

(2) Electrolyte Type

The effect of electrolyte type and cathode processing variables can be readily analyzed in constant current experiments. In tests 84572-3,4, the effect of excess acid was checked. In test 84572-3, 6 ml of 1.5 M $AlCl_3$ plus 0.5 M $MgCl_2$ was used to

Table 2

EFFECT OF ELECTROLYTE PLACEMENT ON TAPE DISCHARGE

Cell No.	Electrolyte Placement	Vol ml	Capacity amp-min	Coulombic Efficiency %
84585-3	Mg	2.5	29.6	49
-5	Mg	2.5	30.8	41
-1	Separator, direct	2.5	29.7	51
-4	Cathode	2.5 +1.0	30.7	30 53
84586-2	Mg	2.5	30.8	52
-3	Separator, direct	2.5	31.0	45
-4	Cathode	2.5	31.2	25

Table 3

EFFECT OF H_2O ON CELL DISCHARGE

0.5 amp/in^2

Cell No.	Electrolyte	Vol ml	Capacity amp-min	Coulombic Efficiency %
84572-6	1.5 $AlCl_3$ 0.5 $MgCl_2$	2.0	28.0	34
-7	1.5 $AlCl_3$ 0.5 $MgCl_2$	4.0	29.6	54
-8	1.5 $AlCl_3$ 0.5 $MgCl_2$	6.0	27.9	69
-9	0.75 $AlCl_3$ 0.25 $MgCl_2$	5.0	26.6	58

activate the tape. The cathode coulombic efficiency was 62%. In test 84572-4, 4 ml of 1.5 M $AlCl_3$ + 0.5 M $MgCl_2$ was used and 2.0 ml of conc. HCl was added after the test was about one-half complete. In this test the efficiency dropped to 49%. Hence, more acid was not required to achieve higher capacities.

The tests in Table 3 show water to be the necessary factor in $AlCl_3 \cdot MgCl_2$ electrolyte.

The effect of various acid and neutral electrolytes have also been analyzed. Some of these results are shown in Table 4.

Table 4

EFFECT OF ELECTROLYTE TYPE ON DISCHARGE EFFICIENCY

0.1 amp/in^2

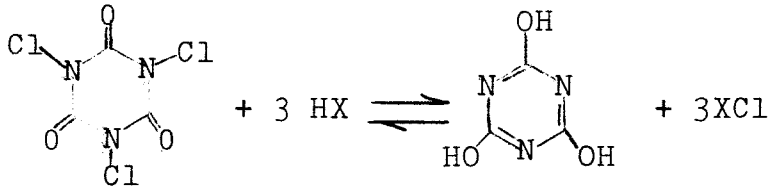
Cell No.	Electrolyte	Vol ml	Capacity amp-min	Coulombic Efficiency %
84575-2	1.5M $AlCl_3$ 0.5M $MgCl_2$	3.0	23.0	47
-3	2M $MgBr_2$	3.0	26.1	40
-4	2M $MgCl_2$	3.0	22.6	39
-5	2M $Mg(ClO_4)_2$	2.5	26.0	37
84579-9	1.5M $AlCl_3$ 0.5M $MgCl_2$	2.5	32.2	50
-4	2M $MgBr_2$	2.5	32.4	50
-6	2M $MgCl_2$	2.5	31.4	45
84582-10	2M $MgBr_2$	2.5	29.1	48
-7	2M $MgCl_2$	2.5	31.2	52
90001-3	2M KHF_2	2.5	30.7	22
84593-5	2M HBF_4*	2.0 +1.0	29.8	25 35

* 0.5 amp/in^2

The results indicate that at low current drain rates there is no advantage in using acid electrolytes, as indicated by coulombic efficiency. Voltages are higher, however, for acidic systems (e.g., Tape 84579-9, $\bar{E}$=2.26 volts and 84579-4, $\bar{E}$=1.96 volts). Because acidic electrolytes cause vigorous anode gassing and cathode decomposition, emphasis was placed on the use of neutral electrolytes this quarter. As seen from Table 4, the differences between $MgBr_2$, $MgCl_2$ and $Mg(ClO_4)_2$ are slight. $MgBr_2$ was chosen as our standard neutral electrolyte.

(3) Acid Fluoride Electrolytes

The use of acidic fluoride ion electrolytes has been suggested to decrease the chemical decomposition of ACL-85, since the equilibrium



would not favor ACL-85 decomposition with HF for HX. HBF_4 and KHF_2 were tested because $HF \cdot H_2O$ was too reactive with magnesium. With KHF_2 there was a pronounced voltage maximum indicating that $MgCl_2$ produced by the reaction was a better electrolyte than the KHF_2 (Table 4). Although there was no maximum with HBF_4 electrolyte, the coulombic efficiency (Tape 84593-5) was inferior to those obtained with $MgBr_2$ and $AlCl_3 \cdot MgCl_2$ electrolytes.

ACL-85 is known to decompose in acidic solution to NCl_3. This process, in addition to Cl_2 formation, might contribute to processing losses and would be accelerated by HF as well as HCl-containing electrolytes. A gas sample was taken of a tape wetted with $AlCl_3 \cdot MgCl_2$ electrolyte. Infrared analysis showed no NCl_3 absorption in this sample. NCl_3 has an absorption peak at 652 cm^{-1} (Ref. 1). The lower limit of detection is not known, however, since the extinction coefficient is not given in the literature.

d. Wetting Agents

Attempts were also made to improve wet-out characteristics of the tapes by including acetone or p-toluenesulfonic acid in the electrolyte to promote wetting of the carbon. Neither reagent inhibited the electrode reaction, and the first test with 1% acetone (Tape 90001-5) indicated a definite improvement in overall performance. However, it does not appear that acetone reduces the electrolyte requirement. p-Toluenesulfonic acid has very little effect on performance (Tape 90001-4,6).

Ref. 1. G. E. Moore and R. M. Badger, J. Am Chem. Soc., 74, 6076 (1952).

Table 5

EFFECT OF WETTING AGENTS ON TAPE DISCHARGE

Cell No.	Electrolyte Additive (to 2M $MgBr_2$)	Vol ml	Capacity amp-min	Coulombic Efficiency %
90001-5	1% acetone	2.5 +1.0	31.0	68 71
-8	1% acetone	2.0 +1.5	27.6	48 62
-10	1% acetone	2.0	25.6	58
-7	2% acetone	2.5 +1.0	27.8	49 58
90005-1	1% acetone	2.1 +1.0	25.6	56 62
-12	none	2.0 +1.0	32.3	43 56
90001-6	1% p-TSA	2.5 +1.0	28.8	54 61
-4	1% p-TSA	1.5 +0.6	27.4	33 62
90005-10	none	1.4 +1.0	30.4	32 61

p-TSA=p-toluenesulfonic acid

Further work on wetting agents is a necessity.

e. Separator Studies

Three separators were tested with our standard $Mg/MgBr_2$/ ACL-85 system. The results shown in Table 6, indicate that the separator material does not affect the efficiency under the reaction conditions.

Table 6

EFFECT OF SEPARATOR ON CELL DISCHARGE

Cell No.	Separator	Vol ml	Capacity amp-min	Coulombic Efficiency %
84590-7	Dynel	2.0	17.9	61
-2	Nylon	2.0	15.3	60
-4	Polypro-pylene	2.0	18.3	62

The use of Dynel separator is being continued.

f. Cathode Composition and Preparation

In the experiments this quarter on tape composition and tape preparation, many variables were changed and simple comparisons are difficult to make. From the data in Table 7, it is evident that decreasing carbon black (Shawinigan acetylene black) content or substituting graphite (Micro-6) for carbon does not noticeably decrease the electrolyte requirement. While certain other variables were also changed during this study, we feel the results illustrate the effect of a change in carbon composition.

Table 7

EFFECT OF CARBON ON Mg/ACL-85 CELL DISCHARGE

$AlCl_3 \cdot MgCl_2$ 0.5 amp/in

Cell No.	SAB %	Graphite %	Vol ml	Capacity amp-min	Coulombic Efficiency %
84553-1	30	0	4.0	38.2	39
84572-2	30	0	2.0	26.7	43
84556-2	18	0	4.0	28.1	33
84556-6	18	0	2.0	23.6	32
84559-6	15	15	4.0	29.8	40
84559-3	15	15	2.0	37.2	17

g. Electrolyte Salts in the Cathode Mix

By adding the hygroscopic salt crystals ($AlCl_3$ $MgCl_2$, $MgBr_2$) to the cathode mix, it was hoped that the activation of the tape would be easier and more complete. In this case, less electrolyte solvent (water) should be needed to activate the tape. Addition of the salts did improve the wetting characteristics of the tapes, although the amount of water required to activate the tapes was not significantly less than when the salts were added in solution. Unfortunately, the ACL-85 cathodes appear to decompose on standing more rapidly when salts are present. Normal loss of active chlorine during tape preparation is about 10%. When $AlCl_3 \cdot H_2O$ (vacuum dried, hydrated crystal) was incorporated into the cathode mix, the active chlorine loss was 33% (Tape 84572). Using $MgCl_2$, active chlorine loss in processing was 16% (Tape 84596), and with $MgBr_2$ the loss was 21% immediately and 32% after 24 hours. Since the results vary because of ACL decomposition, and since no significant improvement in operation was noticed, this approach to wetting improvement has been abandoned. Some representative discharge data are given in Table 8.

Table 8

EFFECT OF THE INCORPORATION OF ELECTROLYTE INTO THE CATHODE MIX

Cell No.	Salt in Tape	Activating Solvent	Capacity amp-min	Coulombic Efficiency %
84597-5	$MgBr_2$	H_2O	25.5	46
-2	$MgBr_2$	H_2O	24.4	64
84596-2	$MgCl_2$	H_2O	30.8	36
-1	$MgCl_2$	H_2O	28.0	43
9005-12	none	$MgBr_2$	32.3	43
84593-1	none	$MgBr_2$	28.6	25
-4	none	$MgBr_2$	26.3	59

h. Fiber Content

Carbon fibers have been used previously in our cathode mixes. They were used primarily to give added mechanical integrity to the tape. The use of better wicking agents, however, should be considered since wetting of the carbon with minimum

quantities of electrolyte is a problem. A comparison was made between carbon, asbestos, and paper pulp. The results are shown in Table 9.

Table 9

EFFECT OF FIBER TYPE ON Mg/ACL-85 CELL DISCHARGE

2M $MgBr_2$

Cell No.	Type of Fiber	Vol ml	Capacity amp-min	Coulombic Efficiency %
90008-2	Carbon	2.0	27.6	35
90005-12	Carbon	2.0	32.3	43
		3.0		56
90007-1	Asbestos	2.0	25.4	38
		3.0		46
90011-11	Paper	2.0	34.1	25
		3.0		61
90011-21	Paper	3.0	33.9	61

The apparent wet-out of tapes with paper pulp was superior to that with carbon fiber or asbestos. With sufficient electrolyte for complete wet-out, the discharge of tapes containing paper pulp was also superior.

i. Gas Evaluation in the Mg/ACL-85 System

Tests using the gas-tight cell (Table 10) designed for methyl formate electrolyte (See IV) were inferior to those using the standard open cell. This indicates that gas polarization is important even with $MgBr_2$ electrolyte. It had been hoped that there would be improvement because of less electrolyte leakage and evaporation.

The tape in the closed cell were activated before cell assembly, and they appeared quite wet, even with 1.0 ml of electrolyte.

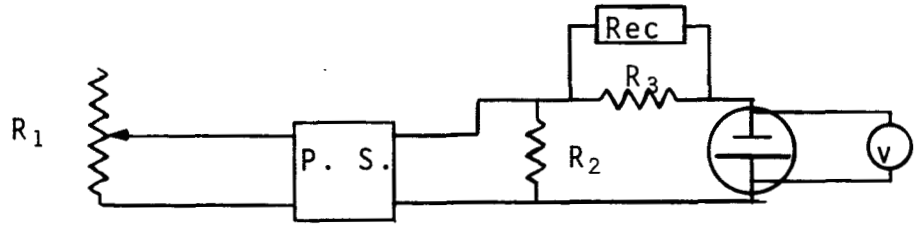
3. Cathode Development - Constant Voltage Test Results With ACL-85 Cathodes

a. Voltage Controlled Discharge

In previous experiments, current control has been employed since it is the most convenient variable to maintain constant. However, since the current collector plates of a dynamic test are at constant voltage, and since the time variable can be converted to distance$\times$speed^{-1}, static voltage-controlled experiments would be more easily correlated with dynamic tests. From the static voltage-controlled experiment, the length of collector, and the position of electrolyte addition in dynamic tests can be evaluated. The mathematical details of a constant voltage discharge are given in the Appendix. Voltage control of the tape drive has been considered for dynamic operation. This could assure continuous, constant voltage discharge, and would allow fast start-up capability and efficient utilization of active materials.

Voltage control of a battery discharge is also the most advantageous method of discharge. The situation can be compared to constant current vs constant voltage coulometry. Using constant voltage, the current drain is greatest when the most material is available. As material is used up, the current decreases. By controlling the voltage externally (by load or tape speed), the greatest percentage of the available power should be obtained. Also less chemical loss should occur with voltage control, since the electrochemical reaction can compete more effectively with the chemical side reactions.

Voltage control of a cell can be achieved by the circuit diagrammed in Figure 1.



R_1 = 500Ω, 10 turn
R_2 ≈ 0.7Ω, 3 amp max
R_3 = 5 amp/50 mv L + N shunt resistor
Rec = Varian G11 recorder set at 30 mv.
P.S. = Harrison Laboratory 6203 Constant Current/Constant Voltage Power Supply
V = Weston voltmeter, 3 volts + 2%

Figure 1. Circuit Diagram

The results of tests using this apparatus are shown in the Appendix as well as in Table 11. The magnitude of the discharge voltage in the 1.7 - 2.2 volt range was found not to be a critical factor in determining the coulombic efficiency.

Table 10

EFFECT OF A CLOSED CELL ON TAPE DISCHARGE

2M $MgBr_2$

Cell No.	Cell Type	Vol ml	Capacity amp-min	Coulombic Efficiency %
90005-2	Closed	1.0	20.6	10
-11	Closed	2.0	29.0	25
-1	Open	2.1	25.6	56
-12	Open	2.0	32.3	43

Table 11

EFFECT OF OPERATING VOLTAGE ON TAPE DISCHARGE

2M $MgBr_2$

Cell No.	volts	Electrolyte vol	Capacity amp-min	Coulombic Efficiency %
90016*-7	1.7	1.5	28.3	48
-5	1.9	1.5	27.6	47
-3	2.0	1.5	28.0	51
-4	2.1	1.5	30.0	36
90021+-3	2.0	1.5	28.9	54
-5	2.1	1.4	27.8	47
-4	2.2	1.4	28.2	45
-7	2.2	1.4	27.1	38
-6	2.4	1.4	25.0	16

* 50 min cut-off, electrolyte added in one batch

\+ 0.06 amp cut-off, electrolyte added in increments

Two (2.0) volts was set as the standard operating voltage. This voltage appeared to be optimum for these tests. Paper pulp was used in the cathode types in place of carbon fibers because of the improvement in tape wet-out characteristics.

b. Aqueous Energy Densities

The results of the best discharge achieved thus far (88 watt-hr/lb) are given in Table 12. This table indicates dramatically the effect of electrolyte weight on the energy density. In this test all of the electrolyte was added at one time.

Table 12

CELL 90011-2-20 Mg/2M $MgBr_2$/ACL-85

Cell Weights	Grams
Anode - Mg Exmet	0.60
Separator - Dynel	0.08
Electrolyte - 2.0 ml of 2M $MgBr_2$	2.60
Cathode - ACL-85	0.83
SAB	0.38
Paper Pulp	0.04
	4.55

Electrical Charactristics - 100 ma cut-off (80 min)

Avg. Amperage	$\bar{I}$ = 0.11 amp/in^2
Voltage	E = 2.0 volts
Area	A = 3.0 in^2
Energy	E = 53.5 watt-min

Energy density = 88 watt-hr/lb

Coulombic capacity (cathode) = 34.7 amp-min
Coulombic efficiency (cathode) = 77%

c. Incremental Addition of Electrolyte

The incremental addition of 2M $MgBr_2$ electrolyte was tested with the constant voltage discharge method using several variations in incremental amounts. With less than 0.7 ml of electrolyte on 3 in^2, practically no discharge was sustained (Table 13).

Table 13

EFFECT OF INCREMENTAL (2.0 VOLTS) ELECTROLYTE ADDITION ON TAPE DISCHARGE

Cell No.	Electrolyte Vol. ml	Capacity amp-min	Coulombic Efficiency,%	Initial Current,amp
90018-3	0.6 initial 1.6 total	29.9	- 83	0.1
-6	0.7 1.3	26.1	- 62	0.6 -
90011-1-12	1.0 1.2	24.4	- 56	1.2 -
-1-10	1.3 1.7	25.8	- 60	1.6 -
90024-4	1.0 1.4	28.5	- 58	0.5 -
-5	1.4 single	28.1	40	1.5

Use of multiple additions improved the coulombic efficiency and energy densities. Cell No. 90018-3 achieved 88 watt-hr/lb (as did cell 90011-2-20, Table 12). The current never exceeded 0.8 ampere in this test.

Acid electrolytes of various concentrations were tested in an attempt to increase the energy densities. However, acid electrolytes appear to offer no advantages over $MgBr_2$ (Table 14). Somewhat higher voltages may be realized in acidic electrolytes, but the electrolyte requirements are higher for the acidic systems. This appears to be due to vaporization of the electrolyte by gassing magnesium.

Table 14

EFFECT OF ACID ELECTROLYTE ON CELL DISCHARGE

Cell No.	Electrolyte	Vol ml	Voltage volts	Capacity amp-min	Coulombic Efficiency %
90011-1-6*	0.5†$AlCl_3$ 1.5 $MgCl_2$	2.0	2.0	30.9	41
90011-1-7*	$MgBr_2$	2.0	2.0	28.0	68
90029-3+	1.5 $AlCl_3$ 0.5 $MgCl_2$	2.5	2.3	27.6	50
-2+	1.5 $AlCl_3$ 0.5 $MgCl_2$	2.5	2.0	29.9	48
-6+	1.5 $AlCl_3$ 0.5 HCl	2.5	2.3	27.6	27
-12+	1.0 $AlCl_3$ 1.0 $MgBr_2$	1.5 2.0	2.3	29.4	28 43
-8+	0.8 $AlCl_3$ 1.2 $MgBr_2$	1.5 2.0	2.0	29.5	22 41
-9+	0.5 $AlCl_3$ 1.5 $MgBr_2$	1.5 2.0	2.0	30.3	14 39
-1+	$MgBr_2$	1.5 2.0	2.0	29.1	57 65

* 50 min cut-off

\+ 0.06 amp cut-off

† Molarity

d. Non-Gassing Electrolytes

In magnesium battery technology, $Mg(ClO_4)_2$ is found to cause the anode to gas less than does $MgBr_2$. Furthermore, saturation of the electrolyte with MgO decreases the gas evolution still further. Mg gassing can be further decreased by using a $LiCrO_4$ inhibitor in $Mg(ClO_4)_2$. With these "non-gassing" electrolytes the tapes appeared to wet with less electrolyte than that used for $MgBr_2$. However, discharges with these electrolytes were not improved (Table 15). More experiments are needed in this area to determine the effectiveness of the "non-gassing"

electrolytes. These advantages in a final design, however, are obvious.

Table 15

EFFECT OF "NON-GASSING" ELECTROLYTES ON TAPE DISCHARGE

2.0 volts

Cell No.	Electrolyte	Vol ml	Capacity amp-min	Coulombic Efficiency %
90021-9	$Mg(ClO_4)_2 \cdot MgO$	1.25	27.1	23
-10	$Mg(ClO_4)_2 \cdot LiCrO_4$	1.25	27.9	37
-11	$MgBr_2$	1.40	27.4	46

4. Cathode Development - Hypochlorite Depolarizers

LiOCl and $Ca(OCl)_2$ tapes were prepared for static evaluation in aqueous electrolytes. The LiOCl tapes burned slowly in air as the trichloroethylene evaporated. We assume this is due to a reaction of Shawinigan acetylene black with the high purity LiOCl (72% LiOCl, Foote Mineral Co.). The $Ca(OCl)_2$ tapes also lost considerable activity during processing [$Ca(OCl)_2$-HTH Olin, 70% $Ca(OCl)_2$, 70% available chlorine]. The chlorine content decreased from 70% to 51%. Tests were run on these tapes using both acidic [$AlCl_3 \cdot MgCl_2$, at 0.1 amp/in.^{2}] and neutral [$MgBr_2$, at 0.1 and 0.5 amp/in.^{2}] electrolytes. The only successful test was with $MgBr_2$ at 0.1 amp/in.^{2}, and the efficiency in this test was only 10%.

Further work in this area depends on finding conducting substrates that do not react with hypochlorite, or catalyze hypochlorite decomposition.

5. Cathode Development - Liquid Cathodes

The use of a liquid cathode material has been suggested as a modified dry tape system. It is presumed that the anode and separator would move, and remove reaction.products from the reaction site. Work was started on the $Mg/MgBr_2/Br_2$ system using the static cell with voltage control. The voltage was set at 2.0 volts, and a 3 to 1 $MgBr_2$(2M)/Br_2 solution was added to a SAB carbon cathode through a platinum screen. The results of this test are shown in Table 16.

Table 16

$Mg/MgBr_2/Br_2$ LIQUID CATHODE TAPE SYSTEM

2.0 volts

Vol ml	Theoretical Capacity amp-min	Actual Capacity amp-min	Coulombic Efficiency %	Watt-hr/lb
1.5	22.2	2.46	11	15
+1.0	36.9	5.54	15	29
+1.0	51.6	8.76	17	36
+1.0	66.3	11.84	18	39

The experiment was halted after four additions of Br_2 solution. It was apparent that the Mg anode was the limiting factor in this experiment. It was almost completely dissolved at the end of the test. The coulombic efficiency would presumably improve further in a dynamic test, since fresh Mg would be supplied continuously and hold-ups of addition by the carbon cathode would not be a significant factor in the long term test. The above energy densities reflect the weight of the tape including cathode electrolyte. In the above test, the electrolytic reaction was fast and a 0.06 amp cut-off was used before addition of more Br_2 solution. The addition of four increments was completed (to 0.06 amp) in 65 min.

Further work in this area would use a dynamic system and a porous carbon block as the cathode. KBr might be used in place of $MgBr_2$ because of greater complexing of Br_2 by KBr. More concentrated Br_2 solutions could then be used.

B. NONAQUEOUS ELECTROLYTE STUDIES

1. Anode Devlopment

Previous half cell and full cell tests have indicated that our 15-mil lithium ribbon anodes show only slight polarizations. For this reason, no change was made in type of anode this quarter. The ribbon was prepared by etching with methanol and scraping until Tape 85363 was prepared, at which time the procedure was changed to the use of lithium as received. We felt that the methanol etch was introducing more problems than it was solving.

2. Nonaqueous Cathode Development

a. ACL-70®, ACL-85®, and CuF_2

This quarter the nonaqueous research program was directed toward minimizing the total cell weight, including electrolyte. This required a gas-tight cell because of the high vapor pressure of methyl formate and it also required a method for adding electrolyte uniformly. The first approach was to use the cell designed for excess electrolyte tests, and then to seal the cell in a polyethylene bag in the glove box. Cathodes of ACL-70, ACL-85, and CuF_2 were discharged in this manner. Results are shown in Table 17.

The ACL-70 discharge was the best of the three systems. However, from the electrolyte volumes required for discharge (especially Tape 85359), it was evident that a more tightly fitted and sealed system was required. Therefore, the new vapor-tight cell described in Section IV. (A) was designed and constructed.

Discharge as a function of electrolyte volume in this cell is shown in Figure 2. The addition of an extra 0.8 ml of electrolyte to the 1.5 ml run (Cell 85371) did not result in any increase in cell capacity. Six cells have been discharged at 0.10 amp/in.^{2} using 1.2 ml of electrolyte. The average capacity above 2.0 volts was 7.75 amp-min, with an average deviation of 0.47 amp-min. The initial capacity of each cell was 20.3 amp-min. It is significant that most of the remaining capacity is in the tape when it is disassembled (8.0 amp-min in Cell No. 85372).

After collecting the above data it was felt that other systems could then be tested and compared in a limited-electrolyte cell. $LiPF_6$ and $LiBF_4$ were prepared as methyl formate solutions. The conductivities were 0.13×10^{-2} ohm^{-1} cm^{-1} for saturated ($\sim$0.3M) $LiPF_6$ and 0.48×10^{-2} ohm^{-1} cm^{-1} for 1M $LiBF_4$. No discharge could be obtained with $LiPF_6$(MF) for Li/ACL-70.

Test Data

Composition: 0.65 g ACL-70
0.15 g SAB
0.05 g C Fiber

Pressure: 10 lb
Area: 3 in.^{2}
Current: 300 ma

Cell No.	Vol. $LiClO_4$(MF)	Sep. Thickness
85371	1.5 ml	6 mils
85372	1.2 ml	6 mils
85380	1.1 ml	3 mils

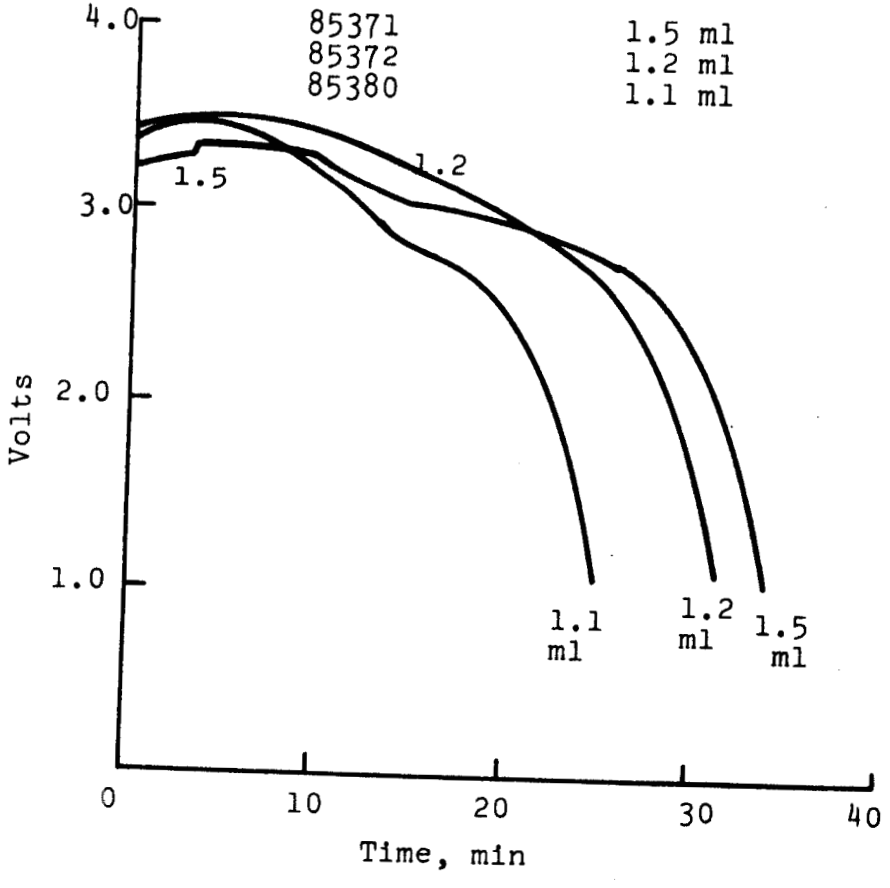


Figure 2. Li/$LiClO_4$(MF)/ACL-70 Tape Cell Discharges at 0.1 Amp/in.^{2}

Table 17

Li/LiClO$_4$(MF) TAPE CELL DISCHARGE - BAG CELL

Ref. No.	Cathode Material	Conductor Material	Cathode Forming Press. lb	Cell Test Press. lb	Current Density amp/in.^{2}	Electrolyte Volume ml	Capacity, amp-min						% Utilization >2.0 v
							Initial	Final	Electrochemical >3.0 v	>2.5 v	>2.0 v	Total	
85365	ACL-70 0.65g	(0.35g SAB) (0.05g C fibers)	15,000	3	0.1	1.8	20.3	7.6	5.4	9.3	9.9	10.8	49
85363	ACL-85 0.65g	(0.17g SAB) (0.17g Pt/C) (0.05g C fibers)	none	30*	0.1	3.0	27.0	2.6	---	3.0	8.4	9.6	31
85359	CuF_2 0.50g	(0.20g Graphite) (0.05g C fibers)	none	24	0.05	1.0	16.5		---	---	---		--
						+0.4			---	2.2	---		--
						+0.1			---	---	6.1		37
						+0.5			---	---	8.4		51

*3 lb was not sufficient to sustain discharge

For $LiBF_4$, voltages of the Li/ACL-70 cell were as good as with $LiClO_4$(MF). However, the capacity was low (5.4 amp-min above 2.0 V at 0.1 amp/in.^{2} discharge rate, Tape 85385..

b. Lithium Dichloroisocyanurate

A lithium salt of dichloroisocyanuric acid was supplied to us by Monsanto Company and was tested in the limited electrolyte cell. The discharge voltage (Cell No. 85393) was lower than for the ACL-70 cells and the cathode efficiency was not improved (37%).

c. Lithium Hypochlorite

LiOCl was tested, using KPF_6 as the electrolyte in methyl formate in the limited electrolyte cell. The cell would not operate above 0.02 amp/in.^{2} and only 0.3 amp-min could be obtained at this drain rate. After disassembling the cell, only 0.3 amp-min of capacity remained. We now suspect that the LiOCl decomposition was not caused by solubility in the electrolyte and reaction with lithium, but rather by decomposition of LiOCl in the cathode mix.

d. Li/ACL-70 Energy Densities

After demonstrating that our Li/ACL-70 results were relatively reproducible, attempts were made to increase the energy density by decreasing the amount of carbon black, carbon fibers, and electrolyte on the ACL-70 system. The results discussed are shown in Figure 3. It was shown that carbon black could be reduced to 0.10 g/0.65 g of ACL-70 (Cell No. 85383). A decrease in current density on this formulation (Cell No. 85389) improved both the voltage and the efficiency, and increased the energy density from 77 to 108 watt-hours/lb. A cursory analysis of the data for calculating energy densities showed that a significant improvement should be made by merely increasing the weight of the cathode while keeping separator and lithium weights constant.

By increasing the cathode weight and keeping the area current density constant, the volume current density was decreased and voltages and cathode capacities were again improved. Cell No. 85388 has a calculated energy density of 144 watt-hours/lb. The data from this cell, our best to date, are shown in Table 18.

This cell was assembled in a dry box. The cathode and separator were wet by syringe and the cell was assembled and pressed with a 10 lb weight, which remained in position during testing. Unfortunately, the variables involved in preparing a cell in this manner are such that as yet we have not been able to reproduce this result. From our attempts to gain reproducibility with this system, it has become evident that many poor discharges

Figure 3. Effect of the Reduction of Carbon Black in Li/ACL-70 Cells.

Cell	Amp-min	A/in²	Eff.	w-hr/lb
85388	40.6	0.05	58	144
85389	20.3	0.05	49	108
85383	20.3	0.10	38	77

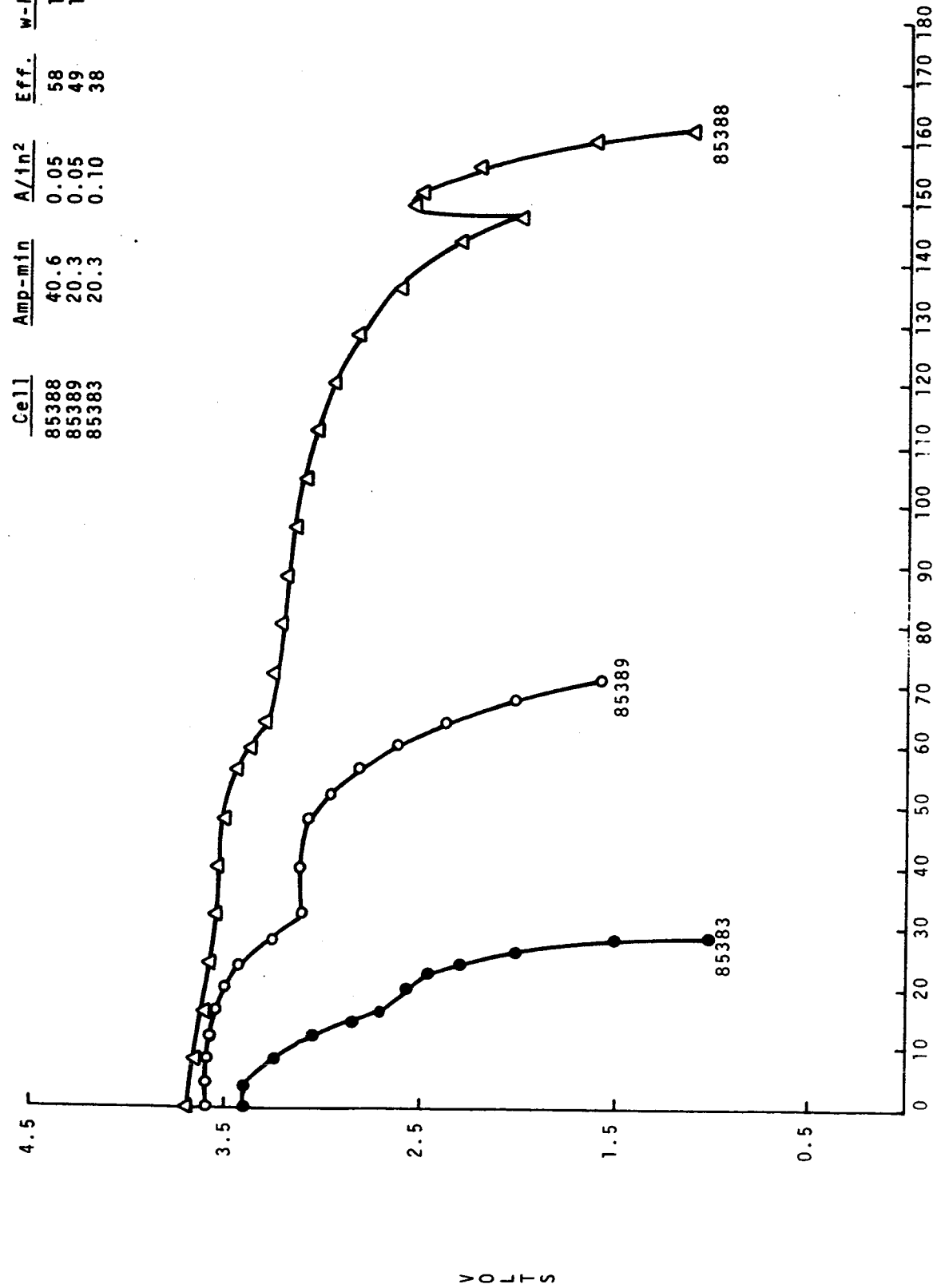


Table 18

CHARACTERISTICS OF CELL 85388

$Li/LiClO_4(MF)/ACL-70$

Cell Weight

Anode - Lithium Ribbon	0.30 g
Separator - 3 mil Polypropylene	0.05
Electrolyte - 1.9 ml 1 M $LiClO_4$ in MF	2.02
Cathode - ACL-70	1.30
SAB	0.20
CF	0.10
	3.97 g

Electrical Characteristics at 2.0 volt cut-off

Ave. voltage	E = 3.18 volts (158 minutes)
Current Density	I = 0.05 amp/in.^{2}
Area	A = 3.0 in.^{2}
Energy	E = 75.3 watt-min

Energy density: 144 watt-hr/lb

Coulombic capacity (cathode) 40.6 amp-min

Coulombic efficiency (cathode) 58%

are caused by poor lithium, separator, and cathode contact. There have been instances in which the lithium, or the cathode, or both were mottled in appearance after discharge. When both are mottled the patterns are the same. In one test (Cell 85399) light and dark areas were cut from the cathode with a cork borer, and each section was analyzed for active chlorine. The white area had, as expected, three times as much undischarged ACL-70 as the dark area. Hence, we are now attempting to prepare more uniform cathodes, and preparing the cell in such a way that it can be activated after assembly. Continuous heavy pressure after activation merely compresses the cathode and squeezes out electrolyte.

Effective utilization of the electrolyte is a significant problem in obtaining a good discharge performance. This problem is evidenced by the further discharge when electrolyte is added after the initial discharge is complete (Cell No. 90806-2). However, to obtain this improvement, the cathode must be scored to facilitate electrolyte penetration since the surface of the cathode becomes hard during discharge. Using this method, cathode efficiency rose from the initial 37% to 57%. This again indicates the importance of cathode formulation and activation variables on the achievement of high energy densities.

III. TASK II. TAPE CELL CONFIGURATION

A. TAPE CELL PREPARATION

1. Static Tape Cells

The fabrication and testing of a tape cell involves many variables. Each variable affects the cell performance in some manner. Since cell reproducibility is necessary if the optimum combination of variables is to be realized, an effort was made to standardize the details of every step in the tape preparation process. The procedures described below relate to the preparation of aqueous static test cells.

a. Pressure

(1) Cathode Formation

The cathode slurry is pressed between a separator and heavy blotting paper in a die at 110 lb/in.^{2} for five seconds.

(2) Cell Formation

Just before a test run, the entire cell is pressed at 200 lb/in.^{2} for two minutes to increase cell contact.

(3) Test Pressure

Test pressure is maintained with spring clamps at 2 lb/in.^{2}.

b. Blending Method

A study of blending methods showed that the exposure of ACL-85 to metal parts lowered the available active chlorine. Mixing, therefore, is now done in a glass apparatus.

c. Carbon Drying

Before use, Shawinigan acetylene black is dried at 135°C/ 1 mm Hg for 24 hours. It is then stored in a desiccator over a drying agent.

d. ACL-85®

ACL-85 is used as received from Monsanto Company. Elevated temperatures or vacuum drying would reduce the active chlorine content.

e. Trichloroethylene

A Baker "Analyzed" grade of trichloroethylene (0.003% water) is now being used as the cathode slurry solvent.

f. Cathode Drying

After pressing the cathodes (see "a" above), the drying process is completed in a vacuum desiccator at 1 mm Hg/room temperature for 16 hours. The cathodes are stored in the desiccator under nitrogen at atmospheric pressure until used. Only eight tapes are prepared at one time in order to avoid long periods before use.

g. Paper Pulp

Two per cent paper pulp is being used in place of carbon fibers to promote cathode wetting. Whatman filter paper is shredded in a Waring Blendor and stored in a desiccator over a drying agent until used.

h. Separators

Two Dynel separators are used in each test cell.

i. Electrolyte

(1) Preparation

The 2M $MgBr_2$ solution is currently being prepared and used until an additional quantity is needed. In the future, a fresh solution will be made weekly.

(2) Addition

Equal amounts of electrolyte are added from a microburet through each of the 15 holes in the static test cell top plate. Since this takes about four minutes, the cell is placed under load before the addition is started in order to reduce ACL-85 activation losses.

2. Dynamic Tape Cells

The apparatus for the preparation of machine-made tapes was reactivated. The piston powered cathode mix extruder would not work with ACL slurries as it had with KIO_4 mixes. Rather than putting a uniform film on the separator prior to rolling, the piston squeezed the solvent from the mix leaving an ACL cake in the piston compartment. Many variations in cathode mix consistency did not solve the problem.

A new method for mix addition was investigated. This method utilizes vibration addition. Preliminary experiments indicate that good tapes can be made using this new addition procedure. Four-foot tapes with mechanical properties equal to those of the previously prepared KIO_4 tapes were made.

B. DYNAMIC TEST APPARATUS

1. Dynamic Testing

Some dynamic tests were made with hand-cast tapes prior to the successful preparation of cathodes by machine. All these tests ended when the tape became too wet and broke under strain, or when so much spent tape remained on the current collector surface that new tape could not make sufficient contact to sustain a discharge. High currents were observed only when the cathode stuck to the current collector.

Best results were obtained when the tape was barely dampened. In this condition the tape remained intact and the collector plate stayed reasonably clean. The stoppage of electrolyte flow through some of the collector holes caused another problem. This led to erratic total currents and to even more erratic changes in partial currents (currents through each collector section). We hope to improve tape-to-collector contact and to eliminate the sloughing off and tape breaking by using a rolling current collector; i.e., letting the tape roll over a rotating disc (see Task III). In this design electrolyte will be fed to the tape only as it enters the collector, unless static tests at constant voltage indicate a decided advantage to incremental additions of electrolyte. A single addition can be more easily controlled.

In one dynamic test, the tape was turned over so that electrolyte would enter through the porous anode. The anode would not wick up the electrolyte, however, and no current could be drawn. This test was made because static results indicated that electrolyte addition through the anode was preferable to addition through the cathode.

2. Static Testing on the Current Collector of the Dynamic Apparatus

Several static tests were run on the dynamic apparatus to evaluate various current collector designs and to determine the necessary test pressures and electrolyte flow rates. The results are listed in Table 19.

The initial results showed that control of current gave data of limited value since many cells failed to operate at potentials above Mg/H_2O values. For this reason, the potential was fixed at 2.0 volts for this series of tests.

The results show that approximately 1 lb/in.^{2} is necessary for adequate tape-current collector contact (Cell 83876, 83878). Vacuum drying of the cathode improved cell performance (Cell 83882), as did elimination of the PVF binder (83881-3, 83882-1).

Table 19

STATIC TESTS ON DYNAMIC TEST APPARATUS - SET AT 2.00 VOLTS - 4.5 $in.^2$ AREA

65% ACL-85 Tape - P-K Blend, Blotting Paper Preparation
Electrolyte 1.5M $AlCl_3$·0.5M $MgCl_2$

Test No.	Notes	Electro-lyte Flow ml/min	Total Flow Time min	Total Volume ml	Test Pressure lb	I_{max} amp	t_{max} min	t >1.5 amp min	t >1.0 amp min	t >0.5 amp min	Notes
83866	(1) (4)	0.73	66.7[x]	48.7	0.65	0.36	22.8	-	-	-	tape dry
83867	(1) (4)	1.44	57.0[x]	82.1	0.65	1.17	15.0	-	5.0-25	5.0-40	tape dry
83868	(1) (4)	2.17	70.0[x]	151.9	0.65	1.35	20.0	-	1.3-35	1.3-50	tape moist and stuck
83869	(1) (4)	3.50	60.8[x]	212.80	0.65	1.47	2.3	-	2.0-15	1.2-40	tape wet
83870	(1) (4)	1.44	45.95[x]	66.17	3.45	1.89	6.0	4.2-12	2.7-20	1.0-30	
83872	(2) (4)	1.44	6.0	8.64	3.45	1.26	6.0	-	2.5-13	1.2-30	10 ml more failed to reactivate
83873	(1) (4)	1.44	6.0	8.64	3.45	1.75	4.7	4.8-6	2.8-9	1.5-20	
83874	(1) (4)	1.44	4.0	5.76	3.45	1.47	6.5	-	1.5-12	1.0-20	
83874-1	(1) (4)	1.44	4.0	5.76	3.45	2.06	4.0	1.5-7	1.2-12	1.0-20	
83875	(1) (4)	1.44	3.0	4.32	10.00	1.22	3.0	-	1.5-5	0.5-15	tape dry
83875-1	(1) (4)	1.44	3.0	4.32	3.45	2.06	3.0	1.0-7	1.5-12	0.6-23	
83876	(1) (4)	1.44	3.0	4.32	1.00	1.45	3.0	-	0.5-12	0.0-25	moist and stuck
83876-1	(1) (4)	1.07	4.0	4.28	1.00	1.35	5.0	-	0.5-12	1.9-27	
83877	(1) (4)	1.07	4.0	4.28	1.00	1.15	6.0	-	5.0-12	1.7-30	dry, not stuck
83878	(3) (4)	1.44	3.0	4.32	1.00	0.36	7.0	-	-	-	
83878-1	(3) (4)	1.44	3.3	4.75	3.45	2.46	4.0	1.0-7	0.9-8	0.5-9	moist and stuck
83879	(2) (4)	1.44	3.0	4.32	3.45	2.22	3.0	2.0-7	1.0-8	1.0-11	moist
83880	(2) (4)	1.44	3.0	4.32	3.45	1.47	2.0	-	1.0-6	1.0-14	unevenly wet
83881	(1) (5)	0.73	6.0	4.38	1.00	0.55	12.0	-	-	7.0-16	dry
83881-1	(1) (5)	0.73	6.0	4.38	3.45	0.87	8.0	-	-	3.0-25	dry
83881-2	(1) (5)	0.73	38.0	27.0	1.00	0.66	8.0	-	-	5.0-15	
83881-3	(3) (5)	0.73	6.0	4.38	3.45	2.20	6.0	4.0-10	2.5-11	2.5-13	stuck
83882	(3) (5) (6)	1.44	3.0	4.32	3.45	0.70	4.0	-	-	2.3-10	
	(7)					1.66	4.0	2.0-5	1.5-8	1.0-15	
	(8)					1.70	3.5	3.0-4	2.0-8	1.5-15	
83882-1	(9) (5)	1.44	10.5	15.1	3.45	1.85	10.0	9.0-12.5	6.7-20	6.0-40	

Underlined variable is principle change from preceding test

(1)Machine made tape ~2.29g(ref. 83864)
(2)Hand-rolled tape coated with graphite(ref. 83865)
(3)Hand-rolled tape-no PVF ~1.3g(ref. 83878)
(4)4-section collector
x Continuous electrolyte feed
(5)Porous cell
(6)Tape dried in vacuum 45 min
(7)" " " " 70 min
(8)" " " " 120 min
(9)Tape hand-rolled without PVF 3.9g

IV. TASK III. SUPPORTING RESEARCH: TEST EQUIPMENT DESIGN AND IMPROVEMENT

A. STATIC TEST CELL HOLDERS

1. Introduction

Different design features are required for aqueous and non-aqueous cell holders. Provisions must be made to remove the hydrogen evolved from an aqueous Mg anode, while a cell using methyl formate solvent must be made vapor tight to keep this low boiling material (32°C) from evaporating.

The static cells used earlier in this work were not designed for use with small amounts of electrolyte, and were inadequate when the limited electrolyte studies were begun this quarter.

Cells capable of utilizing 1 to 2 ml of electrolyte were needed, rather than the 6 ml cells, which were available from the excess electrolyte work. Several cells of different design were constructed to optimize the efficient use of electrolyte with both aqueous and non-aqueous tapes.

2. Aqueous Static Tape Cell Holders

Several designs were considered, and three basic holders were constructed. The first two did not work properly because of design problems that could only be found through actual tests. The third design worked very well and promises to be a very efficient minimum electrolyte cell holder. The top plate is a 1/4x1 3/16x3 inch polyethylene sheet containing 15 equally spaced 1/8-inch diameter holes. Cemented to the bottom of the plate are four equally spaced 1/4x1/16 inch lengthwise runners, which allow uniform pressure to be applied to the cell anode and provide a space for any hydrogen evolved to escape. The bottom plate is a solid polyethylene block fitted with a piece of platinum for the cathode current collector.

The tape cell is assembled between these two plates, which are held together by two 3-pound pressure spring clamps. Several modifications have been proposed to increase the pressure uniformity even more.

3. Non-Aqueous Static Tape Cell Holders

At the beginning of our non-aqueous testing program, we used the cell holders that were also used in our aqueous experi-

ments. As in the aqueous work, these holders were adequate as long as excess electrolyte experiments were being carried out. When limited electrolyte experiments were begun this quarter, it was soon obvious that rapid evaporation of methyl formate solvent was decreasing cell performance. A "bag cell" design was adopted in which the cell was run inside a small polyethylene bag. While this helped, it did not solve the evaporation problem. A new cell holder design was needed.

A vapor tight cell holder was designed and constructed.

The new cell is basically a platinum collector plate in a polypropylene well, with a tight polypropylene cover. The cathode is placed in the well, on the platinum, and it is covered by the separator. Electrolyte is then introduced, the lithium strip is added, and polypropylene cover is placed over the anode. A tab of lithium extends out of the cell for the electrical connection. Pressure is applied to the cell with two 3 pound springs clamps. While a small amount of evaporation is still apparent, the problem has been largely solved. Data reproducibility has been much better since we started using this cell.

The vapor tight cell is pictured in Figure 4.

B. PRESSURIZED NON-AQUEOUS TEST CELL

The evaporation of methyl formate from our vapor tight cell can be reduced further by cooling the cell, or by increasing the atmospheric pressure around the cell. We chose to try the latter method since we felt that pressure had less chance of adversely affecting the electrochemical reaction rate and since cooling inside a dry box presents some problems. A pressurized device, large enough to contain our vapor tight test cell holder, was designed and constructed (Figure 4). Pressures up to 20 psi will be maintained with an argon gas supply. The pressure cell will be tested during the next month.

C. ROLLING CURRENT COLLECTOR FOR THE DYNAMIC APPARATUS

A stationary graphite block is the normal cathode current collector on the dynamic test apparatus. The friction developed as the tape is pulled over the collector necessitates the use of excess electrolyte as a lubricant, and causes crumbling of the cathode. It was felt that a new collector design, one in which most of the friction problems would be eliminated, would overcome several problems. A rolling graphite current collector appeared to have most of the attributes considered necessary. A model has recently been constructed (Figure 5). The rollers are movable and can be placed wherever necessary on the test stand. This device will be tested in the near future.

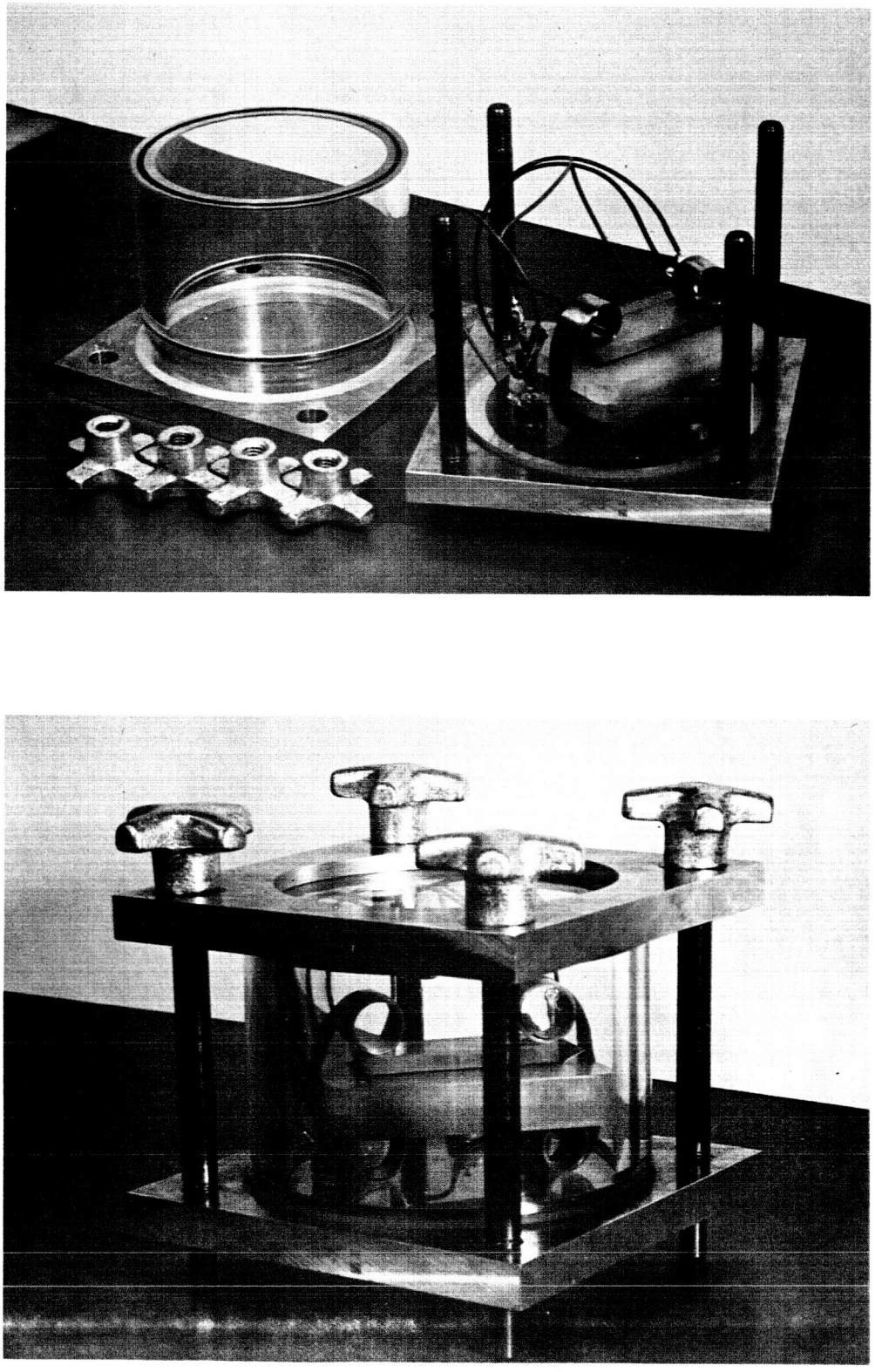


Figure 4. Pressurized Cell for Non-Aqueous Tests.

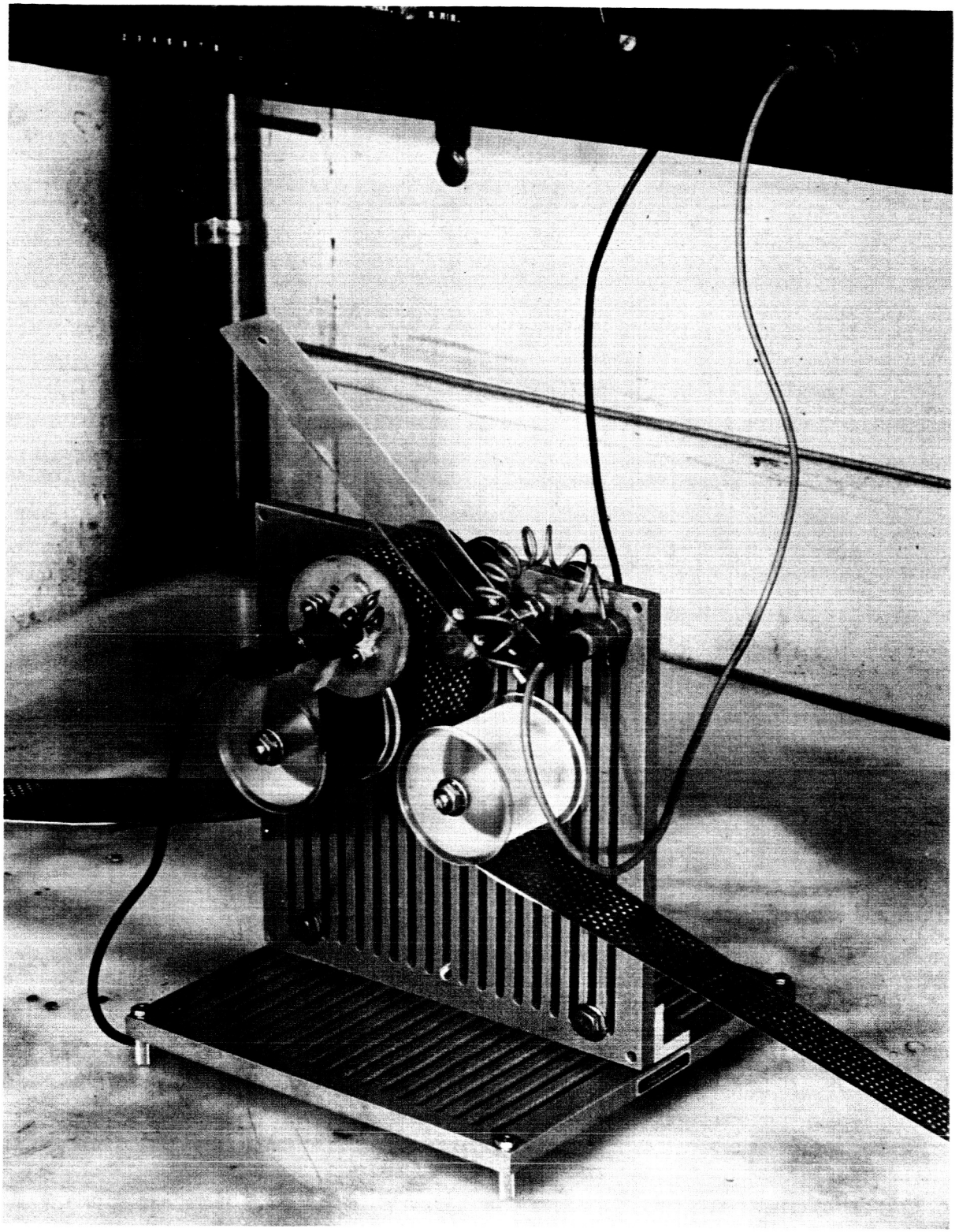


Figure 5. Rolling Current Collector for the Dynamic Test Apparatus.

V. FUTURE PLANS

A. AQUEOUS SYSTEMS

1. The process for the preparation of machine-made cathodes will be improved. A vibrating slurry holder will be substituted for the piston-powered slurry addition device currently used.

2. The new rolling current collector for the dynamic apparatus will be tested with machine-made tapes. An electrolyte feed system for this current collector will be designed.

3. The improtance of polymeric binders to cathode tape integrity will be evaluated.

4. The most important variables in static tape reproducibility studies will be determined.

5. Additional studies will be carried out on the Mg/Br_2 liquid cathode tape system.

B. NON-AQUEOUS SYSTEMS

1. The newly constructed pressurized test cylinder will be evaluated.

2. We will change from a constant current to a constant voltage discharge system. The results of this change will be evaluated on our best system, $Li/LiClO_4(MF)/ACL-70$.

3. We will compare cells prepared from CuF_2 dried with F_2 and Argon.

4. The effect of current collector materials on LiOCl decomposition will be determined.

APPENDIX I

ANALYSIS OF DRY TAPE FUEL CELL DISCHARGE (FLOW SYSTEM BATTERY DISCHARGE)

No analysis has previously been made of the current distribution along the current collector of a dynamic tape device. A current collector has been built that can measure currents in segments of the collector plate. This device can show whether the current collector is sufficiently long and also whether the tape is wet enough when it comes into contact with the collector. Also of interest is the decay of current as a function of distance along the collector, and its dependence on electrochemical and battery variables. For this analysis equations must be derived for the current distribution along the dry tape collector.

The dry tape design of a battery encompasses a constant voltage method of discharge. The time variable is converted to a distance variable along the current collector. The metallic current collectors are at uniform potentials and the current drain through various positions of the collector varies according to the activity of the electrodes sandwiched between them.

It will be assumed in the following calculation that the anode potential does not vary, and that the cathode potential decreases due to concentration polarization, i.e., decrease of active material in the cathode.

We assume that:

$$\frac{dQ}{dt} = -kQ$$

where k is a rate constant (sec^{-1}) that can be potential controlled (e.g., $k=k' \exp \beta\eta$ or $k = k''\eta$, where η is overpotential). We will define Q as moles/cm^2 of electroactive material in a given tape area in the flowing cathode system. Solving the above differential equation gives:

$$Q = Q_0 \exp(-kt)$$

Differentiation with respect to time, and conversion to units of current-density (amp/$in.^2$) gives a segment current time profile of:

$$i_t = nFQ_0k \exp(-kt)$$

With a constant dry tape flow (velocity) of v(cm/sec) the current-distance profile is:

$$i_x = nFQ_0k \exp(-\frac{kx}{v})$$

Thus, we would expect the current to decay logarithmically with time or distance. However, if k/v is small, then the decay would appear linear, as

$$i_x \simeq nFQ_0k \ (1-\frac{kx}{v})$$

The total current can be obtained by integrating over $\underline{x}$ using an arbitrary limit of $\underline{l}$ for the collector length:

$$I = nFQ_0v[1-\exp(-\frac{kl}{v})]$$

For large and small values of kl/v this equation approximates

$$I \simeq nFQ_0v \qquad (\frac{kl}{v} >> 1)$$

$$I \simeq nFQ_0kl \qquad (\frac{kl}{v} << 1)$$

Table A-1

CONSTANT CURRENT STATIC DISCHARGE OF ACL-85 AQUEOUS TAPE CELLS-3 in.^{2}

(Wet Through Exmet Unless Otherwise Noted)

Cell No.	Electrolyte Type	Vol. (ml)	I Amps	Coulombic Efficiency (>1.5 v)	Capacity, amp-min Theoretical	>2.5v	>2.0v	>1.5v	Preparation Variables Pressure lb/in.^{2}	Test Variables Pressure lb/in.^{2}
					Tape 84553					
	(65 parts ACL-85, 29.5 SAB, 2.75 Carbon Fibers, 2.75 PVF, -- P-K Blend)									
1	$AlCl_3 \cdot MgCl_2$	4.0	1.5	39	38.2	-	-	14.4	none	1.0
2	" "	4.0	1.5	34	43.5	-	7.5	15.0	"	4.0
3	" "	4.0	1.5	43	40.0	-	12.0	17.1	"	4.0
4	" "	4.0	1.5	24	42.5	-	-	10.2	0.66 between polyethylene	4.0
5	" "	5.0	1.5	49	42.0	-	6.0	20.6	0.66 " "	8.0
6	" "	4.0	1.5	34	43.3	-	7.5	14.7	0.66 " "	8.0
10	" "	6.0	1.5	56	37.2	-	4.5	21.0	none " blotting paper	8.0
11	" "	6.0	1.5	43	43.6	-	1.5	18.7	3.3 " "	8.0
					Tape 84556					
	(65 parts ACL-85, 15.0 SAB, 2.75 Carbon Fibers, 1.38 PVF -- Waring Blendor)									
1	$AlCl_3 \cdot MgCl_2$	3.0	1.5	21	28.4	-	-	6.5	none	4.0
2	" "	4.0	1.5	33	28.1	-	-	7.5	"	4.0
3	" "	2.4	1.5	33	26.6		1.5	8.8	"	8.0
4	" "	2.0	1.5	32	23.6	-	1.5	7.5	0.66 between polyethylene	6.6
5	" "	1.6	0.5	40	29.2	3.0	9.5	11.8	0.66 between "	6.6
7	" "	2.8	1.0	33	27.0	-	2.0	9.1	3.3 " "	6.6
10	" "	1.0	0.5	19	25.1	-	2.5	4.8	none	5.3
11	" "	1.0	0.5	24	27.3	-	4.5	6.5	3.3 " blotters	8.7
					Tape 84559					
	(65 parts ACL-85, 15 SAB, 15 Graphite, 2.75 Carbon Fibers, 1.38 PVF -- Waring Blendor)									
1	$AlCl_3 \cdot MgCl_2$	6.0	1.5	47	34.8	-	3.0	16.5	none	5.7
2	" "	5.0	1.5	53	31.5	-	6.0	17.1	"	5.3
3	" "	2.0	1.5	17	37.2	-	4.5	6.5	"	5.3
4	" "	5.0	1.5	38	41.5	-	12.0	15.8	2.0 between polyethylene	5.3
6	" "	4.0	1.5	40	29.8	-	1.5	12.0	1.0 " "	5.3
7	" "	3.0	0.5	46	26.7	-	9.5	11.8	1.0 " "	5.3
8	" "	2.0	0.5	47	27.8	4.5	10.5	13.1	5.3 " blotters	5.3
10	" "	2.0	0.5	47	21.4	3.0	8.5	10.2	3.3 " "	9.3
9	" "	2.0	0.5	37	29.4	-	7.5	10.8	3.3 " "	8.3
					Tape 84566					
	(65 parts ACL-85, 29.5 SAB, 2.75 Carbon Fibers, 2.75 PVF, 17 $MgCl_2$ -- Waring Blendor)									
1	$AlCl_3$	2.0	1.5	33	27.4	-	1.5	9.0	none	5.3
2	"	2.0	1.5	37	31.5	-	9.0	11.6	"	8.6
3	"	2.0	0.5	51	32.8	2.0	14.0	16.8	"	8.6
7	"	4.0	0.5	41	33.8	0.5	6.5	14.0	3.3 between blotter	8.6
9	"	2.0	1.5	37	32.6	-	-	12.0	2.0 " "	8.6

Table A-1 (Continued)

CONSTANT CURRENT STATIC DISCHARGE OF ACL-85 AQUEOUS TAPE CELLS-3 in.^{2}

Cell No.	Electrolyte Type	Vol. (ml)	I Amps	Coulombic Efficiency (>1.5 v)	Capacity, amp-min Theoretical	>2.5v	>2.0v	>1.5v	Preparation Variables Pressure lb/in.^{2}	Test Variables Pressure lb/in.^{2}	
					Tape 84568						
	(65 parts ACL-85,29.5 SAB,2.75 Carbon Fibers,2.75 PVF,60 $AlCl_3$,9 $MgCl_2$ -- Waring Blendor)										
2	H_2O	4.0	1.5	25	27.5	-	-	6.9	None	9.3	
4	$AlCl_3 \cdot MgCl_2$	4.0	1.5	21	32.8	-	-	6.8	5.3	9.3	
5	H_2O	4.0	1.5	15	28.0	-	-	4.1	4.0	9.3	
6	H_2O	2.5	0.5	34	26.3	-	5.5	9.0	2.0	9.3	
7	$MgCl_2$	2.0	0.5	38	22.3	-	6.0	8.5	6.7	9.3	
10	H_2O	6.0	1.5	17	25.8	-	-	9.6	2.0	*	
9	H_2O	4.0	0.5	33	26.7	-	6.5	9.2	3.3	*	
					Tape 84572						
	(65 parts ACL-85,29.5 SAB,2.75 Carbon Fibers,2.75 PVF -- Mechanical Stirring of Slurry)										
1	$AlCl_3 \cdot MgCl_2$	4.0	1.5	29	23.4	-	-	6.7	air dried	*	
2	"	2.0	1.5	43	26.7	-	7.5	11.4	"	*	
3	"	6.0	1.5	62	27.0	-	13.5	17.0	"	*	
4	(1)	6.0	1.5	49	24.7	-	10.5	12.0	"	*	
6	$AlCl_3 \cdot MgCl_2$	2.0	1.5	34	28.0	-	4.5	9.6	vacuum dry	9.3	
7	"	4.0	1.5	54	24.6	-	10.5	13.5	"	*	
8	"	6.0	1.5	69	27.9	-	13.5	19.3	"	*	
9	(2)	5.0	1.5	58	26.6	-	9.0	15.4	"	*	
10	$AlCl_3 \cdot MgCl_2$	6.0	1.5	58	25.8	-	12.0	15.0	"	9.3	
5	"	4.0	0.5	63	25.7	11.0	15.2	16.5	air dry	*	
					Tape 84575						
	(65 parts ACL-85,29.5 SAB,2.75 Carbon Fibers,2.75 PVF -- P.K. Blender)										
6	$AlCl_3 \cdot MgCl_2$	4.0	1.5	56	32.6	-	15.0	18.7	None vacuum	*	
8	"	4.0	1.5	47	26.4	-	7.5	12.3	"	4.0	
9	"	4.0	1.5	51	24.7	-	7.5	12.6	"	4.0	
7	"	4.0	1.5	58	21.8	-	10.5	12.6	"	6.0	
10	$MgCl_2$	3.0	0.5	29	16.4	-	2.0	4.6	"	6.0	
4	$MgCl_2$	3.0	0.5	39	22.6	-	2.5	8.8	blotter and vacuum	6.0	
5	$Mg(ClO_4)_2$	3.0	0.5	37	26.0	-	4.5	9.5	"	6.0	
3	$MgBr_2$	3.0	0.5	40	26.1	-	5.5	10.6	"	6.0	
2	$AlCl_3 \cdot MgCl_2$	3.0	0.5	47	23.0	-	10.0	10.9	"	6.0	
1	"	4.0	1.5	44	38.3	-	13.5	16.8	"	6.0	
					Tape 84578						
	(65 parts ACL-85,15 SAB,2.75 Carbon Fibers,2.75 PVF -- P-K Blender)										
2	$MgBr_2$	3.0	0.3	35	29.4	-	2.7	10.3	None vacuum blotter	6.0	
3	"	3.0	0.3	16	36.9	-	2.1	6.0	" " "	6.0	wet before assembly (6n separator)
4	"	3.0	0.3	23	37.8	-	4.2	8.8	" " "	6.0	
6	$AlCl_3 \cdot MgCl_2$	2.5	0.3	46	37.4	-	9.6	17.2	" " "	6.0	

Table A-1 (Continued)

CONSTANT CURRENT STATIC DISCHARGE OF ACL-85 AQUEOUS TAPE CELLS-3 $in.^2$

Cell No.	Electrolyte Type	Vol. (ml)	I Amps	Coulombic Efficiency (>1.5 v)	Capacity, amp-min Theoretical	>2.5v	>2.0v	>1.5v	Preparation Variables Pressure $lb/in.^2$	Test Variables Pressure $lb/in.^2$
					Tape 84579					
				(65 parts ACL-85, 29.5 SAB, 2.75 Carbon Fibers, 2.75 PVF -- P-K Blender)						
2	$MgBr_2$	3.0	0.3	53	24.8	-	7.5	13.0	None, vacuum, blotter	6.0
5	"	3.0	0.3	51	31.6	-	7.5	16.2	" " "	6.0 wet before assembly (on separator)
4	"	2.5	0.3	50	32.4	-	10.2	16.2	" " "	6.0
9	$AlCl_3 \cdot MgCl_2$	2.5	0.3	50	32.2	7.5	11.7	15.9	" " "	6.0
7	"	2.0	0.3	29	30.8	-	7.2	9.0	" " "	6.0
10	"	2.0	0.3	42	33.6	5.7	10.8	13.9	" " "	*
6	$MgCl_2$	2.5	0.3	45	31.4	0.3	5.7	14.3	" " "	6.0
					Tape 84582					
				(65 parts ACL-85, 29.5 SAB, 2.75 Carbon Fibers, 2.75 PVF -- P-K Blender)						
1	$MgBr_2$	2.5	0.3	57	32.3	-	11.1	18.4	110 vacuum blotter	4.0
2	"	2.5	0.3	53	33.4	-	11.7	17.7	110 " "	6.0
3	"	2.5	0.3	55	29.1	-	7.8	16.0	220 " "	4.0
4	"	1.5	0.3	23	29.8	-	4.5	6.8	220 " "	4.0 wet cathode
5	"	2.5	0.3	56	31.4	-	8.4	17.4	330 " "	4.0
6	"	1.5	0.3	23	32.8	-	4.8	7.5	330 " "	4.0 wet before assembly (on separator)
7	$MgCl_2$	2.5	0.3	52	31.2	-	6.3	16.2	1000 " "	4.0
8	$MgBr_2$	2.5	0.3	39	31.3	-	8.0	12.3	1700 " "	4.0
9	$AlCl_3 \cdot MgCl_2$	2.5	1.5	34	29.0	2.2	7.2	14.7	3300 " "	4.0
10	$MgBr_2$	2.5	0.3	48	29.1	-	1.4	3.0	6700 " "	4.0
					Tape 84585					
				(65 parts ACL-85, 29.5 SAB, 2.75 Carbon Fibers, 2.75 PVF -- P-K Blender)						
1	$MgBr_2$	2.5	0.3	51	29.7	-	7.8	15.1	110 vacuum blotter	4.0 wet separator before closing
3	"	2.5	0.3	49	29.6	0.3	12.9	14.5	" " "	4.0 " separator through Mg
4	"	2.5 +1.0	0.3	30 53	30.7	-	7.8 12.0	10.2 16.2	" " "	4.0 " Cathode tape
5	"	2.5	0.3	41	30.8	-	8.1	12.6	" " "	4.0 " separator through Mg
6	"	2.5	0.3	50	30.1	-	8.7	14.9	" " "	4.0 " separator before closing (punched Mg)
7	"	2.5	0.3	38	29.8	-	7.8	11.4	" "	4.0 " " before closing (solid Mg)
8	"	2.5	0.3	43	32.6	-	10.2	13.9	" " "	4.0 " " before closing (punched Mg)

Table A-1 (Continued)

CONSTANT CURRENT STATIC DISCHARGE OF ACL-85 AQUEOUS TAPE CELLS-3 in.^{2}

Cell No.	Type	Vol. (ml)	I Amps	Coulombic Efficiency (>1.5 v)	Capacity, amp-min Theoretical	>2.5v	>2.0v	>1.5v	Preparation Variables Pressure lb/in.^{2}	Test Variables Pressure lb/in.^{2}
					Tape 84585 (Continued)					
9	$MgBr_2$	2.0	0.3	16	32.2	-	3.2	5.1	110, vacuum, blotter	4.0 wet separator before closing (standard Mg
		+1.0		46			6.0	14.7		
					Tape 84586					
					(65 parts ACL-85, 29.5 SAB, 2.75 Carbon Fibers, no PVF -- P-K Blender)					
2	$MgBr_2$	2.5	0.3	52	30.8	0.3	11.7	15.9	110, vacuum, blotter	4.0 wet separator through Mg
3	"	2.5	0.3	45	31.0	-	10.5	13.9	"	4.0 " " before closing
4	"	2.5	0.3	25	31.2	-	4.2	7.8	"	4.0 " cathode tape
5	"	2.0	0.3	29	31.5	-	4.8	9.0	"	4.0 " separator through Mg
6	"	+1.0 3.0	0.3	59 56	30.2	-	9.3	18.6 16.8	"	4.0 2 extra separators (total=4)
8	"	3.5	0.3	0	31.5	-	-	-	"	4.0
9	"	3.0	0.3	52	30.9	-	10.8	16.1	"	4.0 2 extra separators
10	"	3.0	0.3	47	34.1	-	11.7	16.0	54	4.0

Table A-1 (Continued)

CONSTANT CURRENT DISCHARGE OF ACL-85 AQUEOUS TAPE CELLS-3 in.^{2}

Cell No.	Electrolyte Type	Vol. (ml)	I Amps	Coulombic Efficiency (>1.5v)	Capacity, amp-min Theoretical	>2.5v	>2.0v	>1.5v	Preparation Variables Pressure lb/in.^{2}	Test Variables Pressure lb/in.^{2}	Separators	Notes
					Tape 84590							
				(65 parts ACL-85, 29.5 SAB, 2.75 C Fibers, no PVF--P-K Blender, 12% ACL Loss in processing)								
4	$MgBr_2$	2.0	0.3	62	18.3	-	9.0	11.2	110	4	4 polypropylene	
7	"	2.0	0.3	61	17.9	-	7.5	11.0	110	4	3 Dynel	
9	"	1.5	0.3	52	18.8	-	6.0	9.7	110	4	3 Dynel	
		+1.0		63		-		11.8				(3)
2	"	2.0	0.3	60	15.3	-	8.1	9.1	110	4	2 Nylon	
5	$AlCl_3 \cdot MgCl_2$	2.0	1.5	52	17.3	-	6.0	9.0	110	4	4 Polyprop.	
12	$MgBr_2$	1.5	0.3	22	25.7	-	3.7	5.7	110	4	2 Dynel	
		+1.5		56			12.7	14.4				
10	$MgBr_2$	1.0	0.3	37	16.1	-	2.1	6.0	110	4	2 Dynel	(4)
					Tape 84593							
				(65 parts ACL-85, 29.5 SAB, 2.75 C Fibers, no PVF--P-K Blender, 10% ACL loss in processing)								
1	$MgBr_2$	2.0	0.3	25	28.6	-	5.5	7.2	110	4	3 Dynel	
		+1.0		60			15.3	17.1				
2	"	3.0	0.3	56	30.2	-	12.0	16.8	110	4	3 Dynel	
		+1.0		62				18.9				
3	"	2.5	0.3	51	28.7	-	6.9	14.7	110	4	2 Dynel	
4	"	2.5	0.3	59	26.3	-	11.4	15.6	110	4	3 Dynel	
6	$AlCl_3 \cdot MgCl_2$	2.5	0.3	59	29.4	10.5	15.9	17.9	110	4	3 Dynel	
7	" "	2.5	0.3	45	27.9	8.1	11.4	12.6	110	4	3 Dynel	
		+1.0		59			15.0	16.4				(5)
5	HBF_4	2.0	1.5	25	29.8	-	4.5	7.5	110	4	3 Dynel	
		+1.0		35		-	7.5	10.5				
					Tape 84596							
				(65 parts ACL-85,29.5 SAB, 2.75 C Fibers, 38 $MgCl_2$, 16% AC1 loss in processing)								
1	H_2O	2.5	0.3	43	28.0	-	3.0	12.0	110	4	2 Dynel	
2	"	2.0	0.3	36	30.8	-	1.8	11.1	110	4	3 Dynel	
3	"	2.0	0.5	19	28.8	-	1.0	6.5	110	4	2 Dynel	
4	"	2.0	0.3	24	30.7	-	1.5	7.5	110	4	2 Dynel	
					Tape 84597							
				(65 parts ACL-85, 29.5 SAB, 2.75 C Fibers, 74 $MgBr_2$, 21% AC1 loss immediate 32% loss in 24 hrs)								
1	H_2O	2.5	0.3	58	26.3	-	5.1	15.3	110	4	3 Dynel	
3	"	2.5	0.3	64	24.4	-	4.2	15.6	110	4	2 Dynel	
4	"	2.5	0.5	47	27.7	-	3.0	13.0	110	4	2 Dynel	
5	"	2.0	0.3	46	25.5	-	4.5	11.9	110	4	2 Dynel	
6	"	2.0	0.3	43	26.7	-	2.7	11.4	110	4	3 Dynel	

Table A-1 (Continued)

CONSTANT CURRENT STATIC DISCHARGE OF ACL-85 AQUEOUS TAPE CELLS-3 in.^{2}

Cell No.	Electrolyte Type	Vol. (ml)	I Amps	Coulombic Efficiency (>1.5v)	Capacity, amp-min Theoretical	>2.5v	>2.0v	>1.5v	Preparation Variables Pressure lb/in.^{2}	Test Variables Pressure lb/in.^{2}	Separators	Notes
					Tape 90001							
					(65 parts ACL-85, 29.5 SAB, 2.75 C Fibers, P-K Blend, 7% ACl loss in processing)							
3	KHF_2	2.5	0.3	22	30.7	-	4.2	6.9	110	4	2 Dynel	(6)
5	$MgBr_2$ 1%acetone	2.5	0.3	68	31.0	-	5.7	21.0	110	4	"	(7)
		+1.0		71				22.2				
8	"	2.0	0.3	48	27.6	-	9.9	13.2	110	4	"	
		+1.5		62				17.1				
6	$MgBr_2$ 1%pTSA	2.5	0.3	54	28.8	-	7.5	15.6	110	4	"	
		+1.0		61				17.7				
4	"	1.5	0.3	33	27.4	-	5.4	9.0	"	"	"	
		+0.6		62			9.7	17.1				
7	$MgBr_2$·2%acetone	2.5	0.3	49	27.8	-	9.3	13.5	"	"	"	
		+1.0		58				16.2				
10	$MgBr_2$·1%acetone	2.0	0.3	58	25.6	-	11.7	14.8	"	"	"	
										4	"	
					Tape 90005							
					(65 parts ACL-85, 29.5 SAB, 2.75 C Fibers, 4% ACl loss in processing)							
3	$MgBr_2$ 1%acetone	2.5	0.3	42	26.2	-	4.8	10.9	110	4	"	
		+1.0		54				14.2				
1	"	2.1	0.3	56	25.6	-	8.4	14.4	110	4	"	
		+1.0		62				15.9				
11	"	2.0	0.3	25	29.0	-	3.6	7.2	110	4	"	
2	"	1.0	0.3	10	20.6	-	0.9	2.1	110	4	"	(8)
				23			2.4	4.8		hand pressure		(8)
7	1.5$MgBr_2$ 0.5$AlCl_3$	1.8	0.3	51	24.2	2.4	10.5	12.3	110	4	"	
10	$MgBr_2$	1.4	0.3	32	30.4	-	4.2	9.6	110	4	"	
		+1.0		61				18.6	110	4		
9	"	1.6	0.3	33	25.3	-	5.3	8.3	110	4	"	
12	"	2.0	0.3	43	32.3	-	6.3	13.9	110	4	"	
		+1.0		56				18				

Table A-1 (continued)

CONSTANT CURRENT STATIC DISCHARGE OF ACL-85 AQUEOUS TAPE CELLS-3 in.^{2}

Cell No.	Electrolyte Type	Vol. (ml)	I Amps	Efficiency (>1.5v)	Capacity, amp-min Theoretical	>2.5v	>2.0v	>1.5v	Preparation Variables Pressure lb/in.	Pressure lb/in.^{2}	Separators
					Tape 90007						
					(65 parts ACL-85,29.5 SAB, 2.75 Asbestos Fiber, 3% ACL loss in processing--Waring Blendor)						
1	$MgBr_2$	2.0 +1.0	0.3	38 46	25.4	—	4.1	9.6 11.7	110	4.0	2 Dynel
					Tape 90008						
					(65 parts ACL-85, 29.5 SAB, 2.75 Asbestos Fiber--Waring Blendor)						
2	$MgBr_2$	2.0	0.3	35	27.6	-	4.7	9.6	110	4.0	3 Dynel
4	1.5 $MgBr_2$ + 0.5 $AlCl_3$	2.0	0.3	49	27.7	0.9	9.6	13.5	110	4.0	" "
6	1.5 $MgCl_2$ + 0.5 $AlCl_3$	2.0	0.3	43	27.7	5.1	9.6	11.9	110	4.0	" "
					Tape 90011						
					(65 parts ACL-85, 29.5 SAB, 2.75 Paper Pulp--Waring Blendor)						
19	$MgBr_2$	2.0 +1.0	0.3	25 61	34.1	-	6.0 13.2	8.7 21.0	110	4.0	3 Dynel
21	$MgBr_2$	3.0	0.3	61	33.9	-	10.5	20.7	110	4.0	" "

* Screw pressure, light

(1) 4 ml $AlCl_3 \cdot MgCl_2$ + 2 ml HCl (conc.) added at 5 min time

(2) 5 ml of 50% dilution - i.e., 0.75M $AlCl_3$ + 0.25 M $MgCl_2$

(3) Electrolyte added at 1.5 v is recorded separately as + ml

(4) Added electrolyte to separator before closing cell

(5) After test 14% of ACL remained on tape by iodimetric analysis

(6) V-t curve had pronounced maximum

(7) After test 3% of ACL remained on tape

(8) Using closed cell designed for nonaqueous testing

p TSA = p Toluenesulfonic Acid

Table A-2

CONSTANT VOLTAGE STATIC DISCHARGE OF ACL-85 AQUEOUS TAPE CELLS

Cell No.	Electrolyte Type	Vol,ml	Voltage	Coulombic Efficiency	Current(amp)at Time(min) 1	2	5	10	20	50	Final Time min	Final Current amp	Theoretical amp-min capacity	Comments
					Tape 90011-1									
					(65 parts ACL-85, 29.5 SAB, 2.75 C Fiber, Waring Blendor)									
8	2M $MgBr_2$	1.5	2.0	56	2.0	1.5	0.6	0.5	0.3		41	0.04	24.2	(Wet thru Mg unless otherwise noted.)
5	$MgBr_2$	2.0	2.0	66	2.3	1.4	1.1	0.7	0.4		46	0.08	29.7	
3	1.5 $AlCl_3$ 0.5 $MgCl_2$	1.2	2.0	44	2.6	1.6	0.7	0.4	0.1		30	0.07	24.2	
4	$MgBr_2$	1.5	2.0	53	1.7	1.3	0.8	0.5	0.2		50	0.06	26.4	
6	1.5 $MgCl_2$ 0.5 $AlCl_3$	2.0	2.0	41	0.9	0.8	0.5	0.4	0.3		50	0.09	30.9	
7	$MgBr_2$	2.0	2.0	68	2.2	1.8	1.1	0.7	0.4		50	0.06	28.0	
9	$MgBr_2$	1.3	2.0		1.0	0.8	0.6						28.5	
	(added at 6.3 min)	+0.7		68				1.0	0.6		50	0.07		
10	$MgBr_2$	1.3	2.0		1.6	1.0	0.8	0.6					25.8	
	(13 min)	+0.2												
	(16 min)	+0.2		60					0.4		50	0.05		
12	$MgBr_2$	1.0	2.0		1.2	1.1	0.8	0.6					25.4	Wet before assembly
	(13 min)	+0.2		56					0.3		50	0.06		Added thru Mg
1	$MgBr_2$	1.3	2.0		0.8	0.8	0.6	0.4					34.0	Wet before assembly
	(12 min)	+0.8		46					0.4		50	0.17		" " "
11	$MgBr_2$	1.5	2.0	70	2.0	1.5	0.9	0.6	0.4		50	0.06	25.8	" " "
13	$MgBr_2$	1.9	2.0	58	2.0	1.5	0.9	0.6	0.4		50	0.11	30.2	" " "
					Tape 90011-2									
					(65 parts ACL-85, 29.5 SAB, 2.75 Paper Pulp, Waring Blendor, 5% ACL loss in Processing)									
20	$MgBr_2$	2.0	2.0	77	1.3	1.2	1.1	0.8	0.4	0.2	80	0.10	34.7	
23	$MgBr_2$	3.0	2.0	59	2.6	2.1	1.3	0.7	0.3		50	0.15	37.3	
24	$MgBr_2$	2.0	2.0	63	1.8	1.7	1.2	0.8	0.4		50	0.09	33.7	
					Tape 90016									
					(65 parts ACL-85, 30 SAB, 5 Paper Pulp, Waring Blendor, 7% ACL loss in Processing)									
6	$MgBr_2$	1.25	2.0		0.9	0.6	0.4							Wet before assembly
	(6 min)	+0.50		47				0.3	0.2		50	0.13	29.9	Add through Mg
1	$MgBr_2$	1.5	2.0	37	0.9	0.8	0.4	0.3	0.2		50	0.09	27.8	Wet before assembly
2	"	2.0	2.0	59	1.5	1.3	0.8	0.6	0.3		50	0.07	27.0	
3	"	1.5	2.0	51	1.4	1.1	0.7	0.4	0.3		50	0.13	28.0	
4	"	1.5	2.1	36	1.4	1.0	0.5	0.3	0.2		50	0.11	30.0	
5	"	1.5	1.9	47	1.8	1.2	0.8	0.5	0.3		50	0.09	27.6	
7	"	1.5	1.7	48	1.5	1.1	0.7	0.5	0.3		50	0.10	28.3	
					Tape 90018									
					(65 parts ACL-85, 29.5 SAB, 2.75 paper pulp, Waring Blendor)									
2	$MgBr_2$	2.0	2.0	68	2.3	1.8	1.2	0.7	0.4		50	0.10	29.3	
1	"	2.0	2.0	66	2.4	1.8	1.1	0.7	0.4		50	0.10	30.2	
5	"	1.7	2.0	65	2.6	2.0	1.2	0.8	0.4		50	0.07	29.9	
3	"	0.6	2.0		0.1									
	(2 min)	+0.4				0.6	0.4							
	(4 min)	+0.2						0.7	0.3					
	(10 min)	+0.2								0.2	74	0.08	28.3	
	(28 min)	+0.2		83										
4	$MgBr_2$	1.5	2.0	52	1.8	1.4	0.9	0.5	0.3		50	0.10	27.5	
6	$MgBr_2$	0.7	2.0		0.6	0.4							26.1	Cell used with rubber gasket against Mg
	(3 min)	+0.4					0.8	0.6	0.3					
	(28 min)	+0.2		62						0.8	65	0.08		

Table A-2 (continued)

CONSTANT VOLTAGE STATIC DISCHARGE OF ACL-85 AQUEOUS TAPE CELLS

Cell No.	Electrolyte Type	Vol,ml	Voltage	Coulombic Efficiency	Current(amp)at Time(min) 1	2	5	10	20	50	Final Time min	Final Current amp	Theoretical amp-min capacity	Comments
					Tape 90021									
	(65 parts ACL-85, 29.5 SAB, 2.75 paper pulp, Waring Blendor)													
1	$MgBr_2$	0.75	2.0		0.1	0.1								
	(5 min)	0.25					0.5	0.4	0.2				25.4	
	(27 min)	0.10												
	(28 min)	0.15												
	(41 min)	0.10		50							50	0.10		
3	$MgBr_2$	1.00	2.0		0.8	0.7							26.7	
	(5 min)	0.25					0.6							
	(10 min)	0.15						0.4						
	(15 min)	0.10		54					0.3	0.1	83	0.06		
4	$MgBr_2$	1.00	2.2		0.4	0.4							28.9	
	(5 min)	0.25					0.4	0.3						
	(10 min)	0.15		45					0.2	0.1	115	0.06		
5	$MgBr_2$	1.00	2.1		0.3	0.3							28.2	
	(5 min)	0.25					0.3							
	(10 min)	0.15		47				0.4	0.2	0.1	99	0.06		
6	$MgBr_2$	1.00	2.4		0.1	0.1							27.8	
	(5 min)	0.25					0.2							
	(10 min)	0.15		16				0.2	0.1		35	0.06		
7	$MgBr_2$	1.00	2.2		0.5	0.3							25.0	
	(5 min)	0.25					0.2							
	(10 min)	0.15		38				0.3	0.1	0.1	81	0.06		
9	$Mg(ClO_4)_2$ sat'd MgO	1.00	2.0		0.9	0.5							27.1	Tape appears very wet
	(5 min)	0.25		23			0.3	0.2	0.1		46	0.06		
8	$MgBr_2$	1.50	2.0	37	1.4	0.9	0.5	0.3	0.2	0.1	73	0.06	28.0	
10	$Mg(ClO_4)_2$+1%$LiCrO_4$	1.00	2.0		1.2	0.8							27.9	
	(5 min)	0.25		37			0.5	0.4	0.2	0.1	59	0.06		
11	$MgBr_2$	1.00	2.0		0.6	0.4							27.4	
	(5 min)	0.25					0.5							
	(10 min)	0.15		46				0.4	0.2	0.1	67	0.06		
					Tape 90024									
	(65 parts ACL-85 29.5 SAB, 2.75 paper pulp, 4% ACL loss in processing)													
1	$MgBr_2$	1.00	2.0		0.6	0.5							26	
	(5 min)	0.25					0.3							
	(10 min)	0.15		58				0.6	0.3	0.1	65	0.06		
4	$MgBr_2$	1.00	2.0		0.5	0.4							28.5	
	(5 min)	0.25					1.1							
	(10 min)	0.15		55				0.5	0.3	0.1	92	0.06		
5	$MgBr_2$	1.40	2.0	40	1.5	1.1	0.5	0.3	0.2	0.1	75	0.06	28.1	

Table A-2 (continued)

CONSTANT VOLTAGE STATIC DISCHARGE OF ACL-85 AQUEOUS TAPE CELLS

Cell No.	Electrolyte Type	Vol,ml	Voltage	Coulombic Efficiency	Current(amp)at Time(min) 1	2	5	10	20	50	Final Time min	Final Current amp	Theoretical amp - min capacity	Comments
					Tape 90029									
	(67% ACL-85, 30% SAB, 3% Paper Pulp, Waring Blendor, 10% ACL loss in processing)													
1	$MgBr_2$	1.5	2.0	57	1.3	0.9	0.5	0.3	0.2	0.1	114	0.06	29.1	
	(114 min)	+0.5		65							144	0.06		
2	1.5M $AlCl_3$ 0.5M$MgCl_2$	2.5	2.0	48	2.5	2.4	1.3	0.5	0.1		22	0.06	29.9	
3	" " "	2.5	2.3	50	1.6	1.6	1.5	0.5	0.1		28	0.06	27.6	
5	" " "	4.0	2.3	53	2.0	2.1	1.6	0.5	0.1		30	0.06	32.3	
6	1.5M $AlCl_3$ 0.5M HCl	2.5	2.3	27	1.4	2.4	1.6	0.3			18	0.06	27.6	
4	1.5M $AlCl_3$ 0.5M$MgCl_2$	2.5	2.3	37	1.0	1.4	1.2	0.5	0.2		33	0.06	26.5	
8	0.8M $AlCl_3$ 1.2$MgBr_2$	1.5	2.0	(22)	2.6	1.5	0.5	0.2	0.1		23	0.06	29.5	
	(23 min)	+0.5		41						0.1	64	0.06		
9	0.5M $AlCl_3$ 1.5$MgBr_2$	1.5	2.0	(14)	0.9	0.7	0.3	0.1			15	0.06	30.3	
	(15 min)	+0.5		39					0.4	0.1	70	0.06		
10	" " "	1.5	2.0	(23)	1.8	1.2	0.5	0.3	0.1		39	0.06	30.7	
	(39 min)	+0.5		37						0.2	77	0.06		
12	1M $AlCl_3$ 1M $MgBr_2$	1.5	2.3	(28)	1.2	0.8	0.6	0.2	0.1		36	0.06	29.4	
	(36 min)	+0.5		43						0.1	92	0.06		
7	$MgBr_2$	0.6	2.0		0.1								28.8	
	(2 min)	+0.4				0.3								
	(5 min)	+0.5		63			0.8	0.6	0.3	0.1	63	0.06		

Table A-3

NON-AQUEOUS LITHIUM ANODE EXPERIMENTS

Cell No.	Cathode Material Type	%	Separator Thickness mils	Testing Pressure lbs/in²	Current Density A/in²	Electrolyte Volume ml	Capacity, amp-min Initial	Final	>3.5	>3.0	>2.5	>2.0	Coulombic Efficiency % (>2.0v)
85359	CuF_2 (1)	67	3	8	0.05	1.0	16.5						
						+0.4					2.2		
						+0.1						6.1	
						+0.5						8.4	51
85361	ACL-70	72	3	1	0.10	1.8	20.3	13.7		1.8	2.7	3.3	16
85362	ACL-85 (2)	65	3	1	0.10	3.0	27.0	11.6			2.1	3.1	12
85363	ACL-85 (2)	65	3	1	0.10	3.0	27.0	2.6			3.0	5.7	
				10								8.4	31
85364	ACL-70 (3)	64	6	1	0.10	2.5	20.3	6.7		5.4	7.8	8.4	41
85365	ACL-70 (3)	64	6	1	0.10	1.8	20.3	7.6		5.4	9.3	9.9	49
85371	ACL-70 (4)	76	6	3	0.10	1.5	20.3			5.1	8.4	9.5	47
85372	ACL-70	76	6	3	0.10	1.2	20.3	8.0		5.7	7.8	8.7	43
						+0.3					+0.6	+0.9	
85373	ACL-70(5)(6)	76	6	3	0.02	1.2	17.5				2.1		
						+0.8						9.7	55
85374	ACL-70 (6)	76	6	3	0.10	1.2	17.5			3.9	6.9	7.8	45
						+0.5					+0.6	+1.4	
85375	ACL-70 (6)	76	6	3	0.10	1.2	17.5			3.3	6.0	7.2	41
						+0.5					+0.6	+0.9	(46)
85376	ACL-70 (6)	76	6	2	0.10	1.2	17.5			2.4	3.3	3.6	22
						+0.5				+0.9	+2.8	+3.6	(45)
85377	ACL-70 (6)	76	3	3	0.10	1.2	17.5			3.9	6.6	7.8	44
						+0.5						+0.9	50
85378	ACL-70(6)(7)	76	3	3	0.10	1.2	17.5			5.1	7.5	8.1	46
85380	ACL-70 (6)	76	3	3	0.10	1.1	17.5			3.6	5.7	6.9	39
85381	ACL-70	76	3	3	0.10	1.2	20.3			3.6	6.0	6.9	34
						+0.3					+1.8	+2.7	
85382	ACL-70 (8)	76	3	3	0.10	1.5		11.8		3.3	4.8	5.4	26
85383	ACL-70	81	3	3	0.10	1.0	20.3			3.9	6.3	7.8	38
						+0.3					0.6	0.9	
85385	ACL-70 (9)	76	3	3	0.10	1.5	20.3	14.9			1.2	1.8	9
85387	ACL-70	81	3	3	0.05	1.9	40.6		4.5	6.7	8.7	14.3	35
85388	ACL-70	81	3	3	0.05	1.9	40.6		7.5	17.6	21.0	22.2	
				4				13.1			22.8	23.7	58

Table A-3 (Continued)

NON-AQUEOUS LITHIUM ANODE EXPERIMENTS

Cell No.	Cathode Material Type	%	Separator Thickness mils	Testing Pressure lbs/in²	Current Density A/in²	Electrolyte Volume ml	Capacity, amp-min						Coulombic Efficiency % (>2.0v)
							Initial	Final	>3.5	>3.0	>2.5	>2.0	
85389	ACL-70	81	3	3	0.05	1.0	20.3	7.4	3.0	7.5	9.2	10.0	49
85390	ACL-70 (10)	81	3	3	0.05	0.9 +0.3	20.3	11.1		5.2	7.2	7.9	34
85391	ACL-70 (11)	84	3	3	0.05	1.0	20.3	7.7	0.9	4.8	7.6	8.5	45
85392-1	ACL-70	81	3	3	0.05	1.9	40.6		1.1	7.0	11.5	15.9	39
85392-2	ACL-70	81	3	3	0.05	2.0 +0.5	40.6		3.5	6.5	8.5 +0.4	9.6 +1.6	24 28
85393	$LiCl_2CYA$(12)	76	3	3	0.10	1.2	19.7	7.9		0.6	5.7	7.2	37
85394	ACL-70 (11)	84	3	3	0.05	1.9 +0.3	40.6		0.3	3.3 7.3	4.9 8.5	5.0 9.4	12 23
85395	ACL-70	81	3	3	0.05	1.9 +0.3	40.6		1.9	7.0	11.6	13.2 14.4	33 35
85396	ACL-70 (13)	81	6	1-7	0.05	1.9	40.6		0.3	6.7	11.8	16.8	41
85397	CuF_2 (11)	84	3	3	0.10	2.8	43.0			3.0	18.9	22.2	52
85398	ACL-70 (13)	81	3	1-7	0.05	1.9	40.6		4.0	12.6	18.4	20.4	50
85399	ACL-70	81	3	1-8	0.05	1.7	40.6		2.2	11.7	15.8	16.9	52
85400	ACL-70 (14)	81	3	2-7	0.05	1.4	37.8		3.3	7.8	10.9	13.6	36
90801	ACL-70 (14)	81	3	2-5	0.05	1.4	42.0		4.9	10.9	14.7	15.6	37
90802	ACL-70 (15)	81	3	2	0.05	1.5 +0.3	37.0			5.7	8.3 +0.9	10.3 +2.1	28 33
90803	ACL-70	81	3	3-7	0.05	1.9	40.6		0.8	7.5	13.0	15.2	38
90804-1	ACL-70 (10)	74	3	1-3	0.05	2.4	40.6			4.8	12.3	15.9	39
90804-2	ACL-70	81	3	1-3	0.05	1.9	40.6			6.3	11.8	13.0	32
90805-1	ACL-70 (10)	74	3	3	0.05	2.8	63.0			4.8	12.0	14.8	23
90805-2	ACL-70	81	3	3	0.05	2.8	63.0		0.8	8.3	14.8	16.9	27
90806-1	ACL-70 (16)	81	3	3	0.05	1.9	40.6	28.4		2.4	6.5	7.8	19
90806-2	ACL-70 (17) (17)	81	3	3	0.05	1.9 +0.4 +0.3	40.6		0.8	9.9 11.7	11.7 18.9	15.0 21.5 23.0	37 53 57

1) Graphite replaces SAB
2) 1/2 of SAB replaced with Pt/C
3) Cathode compressed at 5000 psi
4) Well type cell from this point down; +0.? ml no improvement
5) Li used as received from this point down
6) Poor ACL-70 Batch Assay ACL=61%
7) Cell fitted with gasket to lessen evaporation from here down
8) 1M $LiBF_4$ in methyl formate
9) $LiClO_4$ (MF)/$B(OCH_3)_3$ = 2/1
10) Graphite added SAB/Graphite=1/1
11) Decrease Carbon Fibers 1/2
12) $LiCl_2CYA$=Lithium dichloroisocyanuric acid
13) Pressure increased during discharge
14) Pre-form cathode with trichloroethylene
15) Pre-formed cathode broken-up
16) Silicone rubber used to attempt to equalize pressure
17) Electrolyte added after scoring cathode

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